The Electrochemical Society . . .

116th Meeting

Columbus
October 18, 19, 20, 21,
and 22, 1959

LOCAL COMMITTEE

General Chairman

C. L. Faust

Vice-Chairman and Hotel Arrangements

C. A. Snavely

Finance

R. W. Hale

Registration

L. D. McGraw

Entertainment

J. H. Winkler

Publicity

R. E. Durtschi

Tours

N. H. Keyser

Transportation

E. M. Sherwood

Ladies' Program

Mrs. F. W. Fink

Columbus Program

THE ELECTROCHEMICAL SOCIETY

One Hundred and Sixteenth Meeting

October 18, 19, 20, 21, and 22, 1959

Sunday through Thursday

CONDENSED TECHNICAL PROGRAM

Monday, October 19, 1959

Battery (Abstracts 1-10) 9:30 A.M.-12:00 M; 2:00-4:30 P.M. (Hall of Mirrors, Deshler Hilton Hotel)

Corrosion—Electronics (Abstracts 42-48) 9:00 A.M.-12:00 M.; 2:00-5:15 P.M. (Ballroom, Deshler Hilton Hotel)

Electro-Organic (Abstracts 123-128) 9:30 A.M.-12:00 M.; 2:00-3:30 P.M. (Yellow Room, Deshler Hilton Hotel)

Electrothermics and Metallurgy (Abstracts 137-144) 9:30-11:30 A.M.; 2:00-4:00 P.M. (Parlor B, Neil House)

Tuesday, October 20, 1959

Battery (Abstracts 11-20) 9:00-11:30 A.M.; 2:30-5:00 P.M. (Hall of Mirrors, Deshler Hilton Hotel)

Corrosion—Electronics (Abstracts 49-54) 9:00-11:30 A.M.; 2:00-4:30 P.M. (Ballroom, Deshler Hilton Hotel)

Electro-Organic (Abstracts 129-136A) 9:00-11:30 A.M;. 2:00-4:00 P.M. (Yellow Room, Deshler Hilton Hotel)

Electrothermics and Metallurgy (Abstracts 145-151) 9:30-11:30 A.M.; 2:00-3:30 P.M. (Parlor B, Neil House)

Wednesday, October 21, 1959

Battery (Abstracts 21-28) 9:30 A.M.-12:00 M.; 2:00-3:30 P.M. (Hall of Mirrors, Deshler Hilton Hotel)

Corrosion—Electronics (Abstracts 55-62) 9:00 A.M.-12:15 P.M.; 2:00-5:15 P.M. (Grand Ballroom, Neil House)

Electrodeposition (Abstracts 63-74) 9:00 A.M.-12:00 M.; 2:00-4:50 P.M. (Yellow Room, Deshler Hilton Hotel)

Electronics—Semiconductors (Abstracts 85-102) 9:00 A.M.-12:15 P.M.; 2:00-5:30 P.M. (Ballroom, Deshler Hilton Hotel)

Thursday, October 22, 1959

Corrosion (Abstracts 29-41) 9:00 A.M.-12:00 M.; 2:00-4:30 P.M. (Grand Ballroom, Neil House)

Electrodeposition (Abstracts 75-84) 9:00-11:30 A.M.; 2:00-4:30 P.M. (Yellow Room, Deshler Hilton Hotel)

Electronics—Semiconductors (Abstracts 103-122A) 9:00 A.M.-12:45 P.M.; 2:00-5:45 P.M. (Ballroom, Deshler Hilton Hotel)

GENERAL INFORMATION

Convention Headquarters will be the Deshler Hilton Hotel, Broad and High Streets, Columbus, Ohio.

Rates per day for rooms at the Deshler Hilton Hotel are: single, $7.50, 9-11, 12-14; double-bed room, $12, 13, 15; twin-bed room, $12-14, 15-18, 18-20; suite, parlor, 1 bedroom, $24-40; parlor, 2 bedrooms, $44.50 and 58.50.

Rates per day for rooms at the Neil House are: single room, $6, 7, 7.50, 8, 9, 10; double-bed rooms, $9.50, 10, 10.50, 11.50, 12.50; twin-bed rooms, $12, 13, 14, 15; suite—parlor, bed room, $16 and up.

Requests for room reservations should be mailed to the hotel. BE SURE to state your intention to attend The Electrochemical Society meeting. Early reservations are advised. If your wife will be attending, be sure to indicate this on your request for reservation.

The Office of the Secretary is in Parlor 216, Ballroom Floor.

Ladies' Headquarters is in Parlor 212, Ballroom Floor.

The Local Committee Office is in Parlor 218, Ballroom Floor.

All technical sessions will be held in the Deshler Hilton Hotel and in the Neil House.

REGISTRATION

The registration desk will be in the East Section of the Ballroom Foyer, Deshler Hilton Hotel. Following is the registration schedule:

Sunday, Oct. 18—3:00 to 9:00 P.M.
Monday, Oct. 19—8:00 A.M. to 6:00 P.M.
Tuesday, Oct. 20—8:30 A.M. to 5:00 P.M.
Wednesday, Oct. 21—8:30 A.M. to 4:00 P.M.
Thursday, Oct. 22—8:30 A.M. to 12:00 M.

Registration fees are:

Members	$ 7.00
Nonmembers	13.00*
Ladies	5.00
Students	2.00
Thursday only	5.00

*If a nonmember fills out a membership application blank and is subsequently elected to membership in the Society, the difference between the nonmember and member registration fee will be applied to his first year's dues.

INFORMATION ABOUT COLUMBUS

Columbus is best known for its industrial diversification and rapid postwar growth. It is strategically located in the industrial heart of the nation so that raw materials and finished products flow freely to and from other industrial centers. Large manufacturing establishments include branches of: North American

Aviation, Inc., Ternstedt Division of General Motors, Westinghouse (Appliance Division), Western Electric, American Brake Shoe, American Blower Company, and Surface Combustion Company. A number of large firms such as Jeffrey Manufacturing Company (mining equipment), Lennox Furnace Company, Seagrave Corporation (firefighting equipment), and Columbus Coated Fabrics Corporation maintain their headquarters and major manufacturing facilities in Columbus. Many smaller industrial firms such as Industrial Nucleonics, Jaeger Machine Company, and Ohio Semiconductors contribute to the diversification and technologic orientation of the industrial community.

Battelle Memorial Institute, the world's largest contract research laboratory, is located in Columbus. The Ohio State University and the Ohio State Research Foundation are well known for scientific and technologic achievements. Capital University is well known for its outstanding liberal arts college, conservatory of music and theological seminary.

As the State Capital, Columbus offers the usual attractions of a metropolitan center in terms of fine restaurants and shopping facilities.

SYMPOSIA

The Battery Division has scheduled symposia on The Leclanché Cell, The Silver-Silver Oxide Electrode, The Lead-Acid Cell, The Nickel Cadmium Cell, and a General Session, all in the Hall of Mirrors, Deshler Hilton Hotel.

The Corrosion Division has scheduled two general sessions, both in the Grand Ballroom Neil House.

The Corrosion and **Electronics Divisions** have scheduled a three-day Joint Symposium on The Surface Chemistry of Metals and Semiconductors, sponsored jointly by the Office of Naval Research and The ECS, on Monday and Tuesday in the Ballroom of the Deshler Hilton Hotel, and on Wednesday in the Grand Ballroom, Neil House.

The Electrodeposition Division has scheduled symposia on Electrode Reactions in Nonaqueous Solutions, Electro- and Chemical-Polishing, and two general sessions, all in the Yellow Room, Deshler Hilton Hotel.

The Electronics Division—Semiconductor Group has scheduled symposia on Elemental Semiconductors, Bulk Properties; Semiconducting Compounds; Process Technology; and Surface Phenomena, Diffusion, all in the Ballroom, Deshler Hilton Hotel.

The Electro-Organic Division has scheduled four sessions, all in the Yellow Room, Deshler Hilton Hotel.

The Electrothermics and Metallurgy Division has scheduled symposia on Refractory Coatings and Less Common Metals, in Parlor B, Neil House.

GENERAL FUNCTIONS

Symposia Chairmen Breakfasts

On **Monday, Tuesday, Wednesday,** and **Thursday,** October 19, 20, 21 and 22, special breakfasts will be

held for symposia chairmen. On the day at which he presides at a session, each chairman should be in the Victorian Room, Deshler Hilton Hotel, at 7:45 A.M.

Monday Buffet Supper and Entertainment

On Monday evening, October 19, an informal buffet supper will be held at the Valley Dale Country Ballroom. Cocktails will be served before supper, and music and entertainment will be provided for the balance of the evening. Special buses will leave the Deshler Hilton Hotel at 5:45 P.M. and will return to the hotel after the entertainment.

Tuesday Palladium Medal Lecture

On Tuesday, October 20, at 5:00 P.M. Professor A.N. Frumkin will deliver the Palladium Medal Lecture in the Ballroom of the Deshler Hilton Hotel.

Tuesday Palladium Medal Reception and Banquet

On Tuesday, October 20, a reception and banquet will be held for the Palladium Medalist, Professor A. N. Frumkin. Cocktails will be served from 6:30 to 7:30 P.M. in the Ballroom Foyer, Deshler Hilton Hotel. Professor Frumkin will receive the Palladium Medal at the banquet. An outstanding speaker will talk on a subject of general interest.

Wednesday Buffet Supper and Get-together

This affair, to be held on Wednesday, October 21, is planned as a gay evening of fun and light entertainment. It will begin at 6:30 P.M. in the Ballroom Deshler Hilton, with cocktails, followed by a buffet supper, music, and an audience participation program. Come and have fun.

PLANT TRIPS

A number of plant trips are being arranged, including a trip to Battelle Memorial Institute's Nuclear Reactor. Final information on the trips will be available later.

LADIES PROGRAM

All the ladies are cordially invited to make Room 212 off the Ballroom Foyer, Deshler Hilton Hotel, their headquarters. Coffee hour will be a daily event from 10 to 11 o'clock except on Wednesday when a special event is scheduled. Members of the Ladies' Committee will be on hand to assist and acquaint guests with the program and to help make the visit to Columbus enjoyable in every way possible.

Sunday, Oct. 18—Registration from 3:00 to 9:00 P.M.

Monday, Oct. 19—Coffee Hour and Get Acquainted Meeting in Room 212 from 10:00 to 11:00 A.M. Buses will leave the Hotel at 12:30 P.M. to take the ladies to the Ohio Union on the Ohio State University campus, where lunch will be served. After the luncheon

there will be a talk on flower arrangements, followed by optional tours of the Ohio Union, Ohio State Museum, and the Department of Horticulture. Walking shoes are recommended.

Monday evening, Oct. 19, the ladies are invited to attend a Buffet Supper at the Valley Dale Country Ballroom (see General Functions).

Tuesday, Oct. 20—Coffee Hour in Room 212 from 10:00 to 11:00 A.M.

Tuesday afternoon, the ladies are invited to attend a luncheon at 1:00 P.M. at the Columbus Athletic Club, followed by a program of folk lore songs by Mrs. Anne Laylin Grimes.

Tuesday evening, the ladies are invited to attend the reception and banquet at 6:30 P.M. in the Ballroom (see General Functions).

Wednesday, Oct. 21—At 10:00 A.M., buses will leave the Deshler Hilton Hotel to take the ladies to Ilonka's Provincial House, where they will be served a complimentary brunch. A bus tour to the Ericson Glass Works at Bremen, Ohio, will start from there and occupy most of the afternoon.

Wednesday evening, the ladies are invited to attend the Buffet Supper and Informal Get-Together beginning at 6:30 P.M. in the Ballroom (see General Functions).

Thursday, Oct. 22—Coffee Hour in Room 212 from 10 to 11:00 A.M. All ladies are cordially invited to attend an informal sherry party at 11:30 A.M. at the home of Mrs. F. W. Fink, 1900 North Devon Road, Columbus, Ohio. Mrs. Fink is the Chairman of the Ladies Committee.

BOARD AND COMMITTEE MEETINGS

Sunday, Oct. 18, 2:00 P.M.—Meeting of the **Board of Directors** in Room 307, Deshler Hilton Hotel.

Sunday, Oct. 18, 7:30 P.M.—Meeting of the **Investment Advisory Panel** in Private Dining Room 10.

Monday, Oct. 19, 4:30 P.M.—Meeting of the **Council of Local Sections** in Room 307.

Monday, Oct. 19, 5:00 P.M.—Meeting of the **Sustaining Membership Committee** in Private Dining Room 10.

Monday, Oct. 19, 5:00 P.M.—Meeting of the **Editorial Staff of the Journal** in Private Dining Room 12.

Wednesday, Oct. 21, 5:00 P. M.—Meeting of the **Membership Committee,** in Private Dining Room 10.

LUNCHEONS AND BUSINESS MEETINGS OF DIVISIONS

Electro-Organic Division Luncheon and Business Meeting, Tuesday, Oct. 20, 12:15 P.M. in Room 307, Deshler Hilton Hotel.

Battery Division Luncheon and Business Meeting, Tuesday, Oct. 20, 12:15 P. M. in Private Dining Rooms 10, 11, and 12, Deshler Hilton Hotel.

Electrodeposition Division Luncheon and Business Meeting, Wednesday, Oct. 21, 12:15 P.M. in Spanish Room, Deshler Hilton Hotel.

Corrosion Division News Luncheon and Business Meeting, Thursday, Oct. 22, 12:15 P.M. in Mezzanine Parlor 4, at the Neil House.

COST OF VARIOUS FUNCTIONS

(other than registration)

Please buy tickets as early as possible.

Symposia Chairmen Breakfasts-Standard menu prices	
Monday Buffet Supper and Entertainment	$5.25
Tuesday Palladium Medal Reception and Banquet, men	7.50
ladies	6.00
Wednesday Buffet Supper	4.50
Division Luncheons	3.50
Transportation for plant visits	1.00
Ladies' Program	
Monday luncheon	2.00
Tuesday luncheon	2.00
Wednesday transportation to Bremen	1.00

JOURNALS

Copies of the July, August, September, and October issues of the "Journal of The Electrochemcial Society" will be available at the registration desk. The price of a single copy of the Journal is $1.25 to members and $1.75 to nonmembers.

SPRING MEETING
at the
La Salle Hotel
CHICAGO, ILLINOIS
May 1, 2, 3, 4, and 5, 1960

Sessions probably will be scheduled on:

Electric Insulation
Electronics (including Luminescence and Semiconductors)
Electrothermics and Metallurgy
Industrial Electrolytics
Theoretical Electrochemistry

Abstracts for the Chicago Meeting (*not exceeding 75 words in length*) must reach Society Headquarters, 1860 Broadway, New York 23, N.Y., *not later than January 4, 1960* to be included in the Program.

Please indicate on the abstract for which Division's symposium the paper is to be scheduled and underline the name of the author who will present the paper at the Chicago Meeting.

EMPLOYMENT POSTERS

Companies which desire to recruit employees at the Columbus Meeting will have posters to this effect on a board near the registration desk. Companies are requested to confine their announcements to this board.

NOTE

In the Index to Authors, the abstract number is used. Address of each author is included with the abstract.

EXTENDED ABSTRACTS

The Battery Division is making available an extended abstract booklet containing 1000-word abstracts of most of the papers presented at the Division's Columbus Meeting. Copies of the booklet may be obtained from C. H. Clark, 34 Pleasant Place, Deal, N. J., after Sept. 15 for $2.00 each.

DISCUSSION

No recordings will be made of oral discussions. Those contributing to the discussion of a paper and desiring their remarks to be published will be supplied by the symposium chairman with a printed form on which any discussion may be written. These forms should be given to the Secretary-Treasurer of the Division or to the Managing Editor of the *Journal* after the session; they can be mailed to the Managing Editor of the *Journal*, 1860 Broadway, New York 23, N. Y. The discussion will then be referred to the author for reply. Publication of the discussion and the comments of the author depends on publication of the article in the Journal.

Written discussion should be submitted within two months following publication of the article in the *Journal*. A Discussion Section is published semiannually in the *Journal*.

Technical Program

BATTERY

Monday, October 19, 1959
The Leclanché Cell
with J. C. White presiding

(HALL OF MIRRORS, Deshler Hilton Hotel)

9:30 A.M.—"Study of the Recuperation Reaction in the Leclanché Dry Cell" by M. P. Korver, R. S. Johnson, and N. C. Cahoon (Abstract No. 1)

10:00 A.M.—"A Method of Increasing the Service Life of Leclanché Dry Cells" by H. J. DeWane (Abstract No. 2)

10:30 A.M.—"A Method for Evaluating Battery Grade Manganese Dioxide" by Friedrich Kornfeil (Abstract No. 3)

MEETING ROOM SCHEDULES

Meeting	Monday		Tuesday		Wednesday		Thursday	
	A.M	P.M.	A.M	P.M.	A.M	P.M.	A.M	P.M.
Battery	B	B	B	B	B	B		
Corrosion							D	D
Corrosion—Electronics	A	A	A	A	D	D		
Electrodeposition					C	C	C	C
Electronics—Semiconductors					A	A	A	A
Electro-Organic	C	C	C	C				
Electrothermics and Metallurgy	E	E	E	E				

Deshler Hilton Hotel: A = Ballroom; B = Hall of Mirrors; C = Yellow Room.
Neil House: D = Grand Ballroom; E = Parlor B.

11:00 A.M.—"An Improved Iodide Method for the Analysis of Manganese Dioxide" by F. J. Krivanek and N. C. Cahoon (Abstract No. 4)

11:30 A.M.—"Impedance of Leclanché Cells" by R. J. Brodd and H. J. DeWane (Abstract No. 5)

Battery (cont'd)
Monday, October 19, 1959
The Leclanché Cell (cont'd)
with W. S. Herbert presiding

(HALL OF MIRRORS, Deshler Hilton Hotel)

2:00 P.M.—"The Overpotentials of Some Oxide Electrodes and the Discharge Mechanism" by W. C. Vosburgh, J. H. DeLap, and H. B. Mark, Jr. (Abstract No. 6)

2:30 P.M.—"Gravity Field Effects on Zinc Anode Discharge in Alkaline Media" by M. Eisenberg and H. F. Bauman (Abstract No. 7)

3:00 P.M.—"Magnesium Primary Cells with Perchlorate Electrolytes" by G. S. Lozier and C. K. Morehouse (Abstract No. 8)

3:30 P.M.—"A Synchronized Square Wave Generator and Switching Unit for Electrochemical Studies" by N. C. Cahoon and H. F. Schaefer (Abstract No. 9)

4:00 P.M.—"Sine Wave Pulse Current Tester for Batteries" by K. Kordesch and A. Marko (Abstract No. 10)

Battery (cont'd)
Tuesday, October 20, 1959
The Silver-Silver Oxide Electrode
with Walter Hamer presiding

(HALL OF MIRRORS, Deshler Hilton Hotel)

9:00 A.M.—"The Silver/Silver-Oxide Electrode System" by B. D. Cahan, J. B. Ockerman, R. Amlie, and P. Rüetschi (Abstract No. 11)

9:30 A.M.—"Oxides on the Silver Electrode" by C. P. Wales and Jeanne Burbank (Abstract No. 12)

10:00 A.M.—"The Stability and Solubility of AgO in Alkaline Solutions" by T. P. Dirkse and Brandon Wiers (Abstract No. 13)

10:30 A.M.—"The Oxidation of the Silver Electrode in Alkaline Solutions" by T. P. Dirkse (Abstract No. 14)

11:00 A.M.—"The Effect of Cycling on the Silver Electrode" by T. P. Dirkse and J. De Vries (Abstract No. 15)

12:15 P.M.—Battery Division Luncheon and Business Meeting in Private Dining Rooms 10-12, Deshler Hilton Hotel.

Battery (cont'd)
Tuesday, October 20, 1959
General Session
with C. H. Clark presiding

(HALL OF MIRRORS, Deshler Hilton Hotel)

2:30 P.M.—"Factors Affecting the Photogalvanic Behavior of Silver-Silver Halide Electrodes" by S.

Zaromb, M. Lasser, and F. Kalhammer (Abstract No. 16)

3:00 P.M.—"Silver Halide Photogalvanic Batteries" by S. Zaromb, M. Lasser, and L. Bockstie (Abstract No. 17)

3:30 P.M.—"Burgess Mercury Cells" by T. H. Loverude (Abstract No. 18)

4:00 P.M.—"Investigation of the Electrochemical Characteristics of Organic Compounds, V. Heterocyclic Nitro Compounds" by R. Glicksman and C. K. Morehouse (Abstract No. 19)

4:30 P.M.—"Investigation of the Electrochemical Characteristics of Organic Compounds, VI. Aromatic Hydroxy, Aromatic Amine, and Aminophenol Compounds" by R. Glicksman (Abstract No. 20)

5:00 P.M.—Palladium Medal Address by A. N. Frumkin in the Ballroom, Deshler Hilton.

Battery (cont'd)
Wednesday, October 21, 1959
The Lead-Acid Cell
with E. J. Ritchie presiding
(HALL OF MIRRORS, Deshler Hilton Hotel)

9:30 A.M.—"The Principal Source of α-PbO_2 in the Positive Plate Material of the Lead-Acid Battery" by V. H. Dodson (Abstract No. 21)

10:00 A.M.—"The PbO_2 Electrode" by R. T. Angstadt, B. D. Cahan, and P. Rüetschi (Abstract No. 22)

10:30 A.M.—"The Microscopy of the Effect of Environmental Changes on the Forming Process" by A. C. Simon and E. L. Jones (Abstract No. 23)

11:00 A.M.—"The Microscopy of the Forming Process in the Positive and Negative Plate" by A. C. Simon and E. L. Jones. (Abstract No. 24)

11:30 A.M.—"Internal Resistance of Lead-Acid Cells; Submarine Battery Size" by J. J. Lander and E. E. Nelson (Abstract No. 25)

Battery (cont'd)
Wednesday, October 21, 1959
The Nickel Cadmium Cell
with A. Fleischer presiding
(HALL OF MIRRORS, Deshler Hilton Hotel)

2:00 P.M.—"Nickel-Cadmium Cell Electrodes" by A. J. Salkind and P. F. Bruins (Abstract No. 26)

2:30 P.M.—"Investigations on the Reaction Mechanism of the Nickel-Cadmium Cell" by S. Uno Falk (Abstract No. 27)

3:00 P.M.—"Sealed Nickel-Cadmium Cells Containing Palladium" by H. N. Seiger (Abstract No. 28)

CORROSION

Thursday, October 22, 1959
with R. T. Foley presiding
(GRAND BALLROOM, Nell House)

9:00 A.M.—"The Attack of Copper-Gold, Silver-Gold, Copper-Nickel, and Silver-Copper Alloys by Sulfur

at Elevated Temperatures" by B. D. Lichter and Carl Wagner (Abstract No. 29)

9:25 A.M.—"Anodic Oxidation of High-Purity Aluminum in Water" by W. E. Tragert and J. R. Rairden (Abstract No. 30)

9:50 A.M.—"The High-Temperature Aqueous Corrosion Resistance of the Uranium-5% Zirconium-1½% Niobium Alloy" by J. E. Draley, Sherman Greenberg, and W. E. Ruther (Abstract No. 31)

10:15 A.M.—"Evidence for a Logarithmic Oxidation Process for Stainless Steel in Aqueous Systems" by Milton Stern (Abstract No. 32)

10:40 A.M.—"Passivating Inhibitors" by A. C. Makrides and Milton Stern (Abstract No. 33)

11:05 A.M.—"Dissolution of Metals in Aqueous Acid Solutions" by A. C. Makrides (Abstract No. 34)

11:30 A.M.—"Potential Behavior of a Uranium Electrode as a Function of Partial Pressure of Oxygen" by J. W. Ward and J. T. Waber (Abstract No. 34A)

12:15 P.M.—Corrosion Division News Luncheon and Business Meeting in the Mezzanine Parlors, Neil House.

Corrosion (cont'd)

Thursday, October 22, 1959

with M. A. Streicher presiding

(GRAND BALLROOM, Neil House)

2:00 P.M.—"An Electrochemical Study of Passive Films on Fe-Cr Alloys" by D. Caplan, A. Harvey, and M. Cohen (Abstract No. 35)

2:20 P.M.—"Effect of Surface Preparation on Air-formed Films on Iron" by C. D. Stockbridge, P. B. Sewell, and M. Cohen (Abstract No. 36)

2:40 P.M.—"Nitric-Hydrofluoric Acid Pickling of Zirconium Alloys" by M. A. DeCrescente, P. F. Santoro, A. S. Powell, and R. H. Gale (Abstract No. 37)

3:00 P.M.—"An Investigation of Chemical Variables Affecting the Formation of Films on Copper in Aqueous Solutions" by W. H. Davenport, V. F. Nole, and W. D. Robertson (Abstract No. 38)

3:20 P.M.—Ten-minute intermission

3:30 P.M.—"Dissolution of Single Crystals of Copper in Aqueous Ethylenediamine" by L. H. Jenkins (Abstract No. 39)

3:50 P.M.—"The Electrochemical Behavior and Passivity of Titanium" by Milton Stern and Herman Wissenberg (Abstract No. 40)

4:10 P.M.—"The Influence of Noble Metal Alloy Additions on the Electrochemical and Corrosion Behavior of Titanium" by Milton Stern and Herman Wissenberg (Abstract No. 41)

CORROSION—ELECTRONICS

Joint Symposium
Monday, October 19, 1959
The Surface Chemistry of Metals and Semiconductors
General Aspects of the Chemistry and Physics of Surfaces
Sponsored Jointly by the Office of Naval Research and The ECS
with H. C. Gatos presiding
(BALLROOM, Deshler Hilton Hotel)

9:00 A.M.—Introductory Remarks by H. C. Gatos
9:15 A.M.—"Introduction to the Physics and Chemistry of Surfaces" by W. H. Brattain (Abstract No. 42)
10:15 A.M.—"Metal Surfaces" by H. J. Juretschke (Abstract No. 43)
11:00 A.M.—Fifteen-minute intermission
11:15 A.M.—"Semiconductor Surfaces" by Paul Handler (Abstract No. 44)

Corrosion—Electronics
Joint Symposium (cont'd)
Monday, October 19, 1959
The Surface Chemistry of Metals and Semiconductors (Cont'd)
General Aspects of the Chemistry and Physics of Surfaces (cont'd)
Sponsored Jointly by the Office of Naval Research and The ECS
with J. W. Faust, Jr., presiding
(BALLROOM, Deshler Hilton Hotel)

2:00 P.M.—"Clean Surfaces" by H. E. Farnsworth (Abstract No. 45)

Nature and Behavior of Imperfect Surfaces

2:45 P.M.—"The Role of Imperfections in Surface Reactions" by N. Cabrera (Abstract No. 46)
3:30 P.M.—Fifteen-minute intermission
3:45 P.M.—"Damaged Surface Layers—Semiconductors" by T. M. Buck (Abstract No. 47)
"Damaged Surface Layers—Metals" by L. E. Samuels will be included in the published Proceedings but will not be presented at this symposium.
4:30 P.M.—"Effect of Imperfections on Dissolution" by J. J. Gilman (Abstract No. 48)

Corrosion—Electronics
Joint Symposium (cont'd)
Tuesday, October 20, 1959
The Surface Chemistry of Metals and Semiconductors (cont'd)
Electrode Behavior of Metals and Semiconductors
Sponsored Jointly by the Office of Naval Research and The ECS
with Milton Stern presiding
(BALLROOM, Deshler Hilton Hotel)

9:00 A.M.—"Metal and Semiconductor Electrode Processes" by H. Gerischer (Abstract No. 49)

9:45 A.M.—"Experimental Techniques for the Study of Semiconductor Electrodes" by J. F. Dewald (Abstract No. 50)
10:30 A.M.—Fifteen-minute intermission
10:45 A.M.—"Electrode-Solution Interfaces" by H. J. Engell (Abstract No. 51)

Corrosion—Electronics
Joint Symposium (cont'd)
Tuesday, October 20, 1959
The Surface Chemistry of Metals and Semiconductors (cont'd)
Etching Processes
Sponsored Jointly by the Office of Naval Research and The ECS
with C. V. King presiding

(BALLROOM, Deshler Hilton Hotel)

2:00 P.M.—"Electrolytic Attack of Metals" by Paul Lacombe (Abstract No. 52)
2:45 P.M.—"Electrolytic Etching of Semiconductors" by D. R. Turner (Abstract No. 53)
3:30 P.M.—Fifteen-minute intermission
3:45 P.M.—"The Etching of Metals and Semiconductors" by J. W. Faust, Jr. (Abstract No. 54)
5:00 P.M.—The Palladium Medal Address by A. N. Frumkin in the Ballroom, Deshler Hilton Hotel.

Corrosion—Electronics
Joint Symposium (cont'd)
Wednesday, October 21, 1959
The Surface Chemistry of Metals and Semiconductors (cont'd)
Surface Reactions in Liquid Media
Sponsored Jointly by the Office of Naval Research and The ECS
with M. A. Streicher presiding
(GRAND BALLROOM, Nell House)

9:00 A.M.—"Kinetics of Dissolution Processes" by Norman Hackerman (Abstract No. 55)
9:45 A.M.—"Electrochemistry of Dissolution Processes" by J. V. Petrocelli (Abstract No. 56)
10:30 A.M.—Fifteen-minute intermission
10:45 A.M.—"Dissolution of Metals" by C. V. King (Abstract No. 57)
11:30 A.M.—"Dissolution of Semiconductors" by H. C. Gatos (Abstract No. 58)

Corrosion—Electronics
Joint Symposium (cont'd)
Wednesday, October 21, 1959
The Surface Chemistry of Metals and Semiconductors (cont'd)
Surface Reactions in Gaseous Media
Sponsored Jointly by the Office of Naval Research and The ECS
with E. A. Gulbransen presiding

(GRAND BALLROOM, Nell House

2:00 P.M.—"Adsorption and Chemisorption" by M. Boudart (Abstract No. 59)
2:45 P.M.—"Chemisorption and Catalysis" by R. P. Eischens (Abstract No. 60)
3:30 P.M.—Fifteen-minute intermission
3:45 P.M.—"On the Mechanism of the Oxidation of Metals" by K. Hauffe (Abstract No. 61)
4:30 P.M.—"The Influence of Crystal Orientation on Oxidation of Metals" by A. T. Gwathmey and K. R. Lawless (Abstract No. 62)

ELECTRODEPOSITION

Wednesday, October 21, 1959
Electrode Reactions in Nonaqueous Solutions
with Abner Brenner presiding

(YELLOW ROOM, Deshler Hilton Hotel)

9:00 A.M.—"Anodic Methoxylation" by K. E. Kolb, T. W. Lippincott, and C. L. Wilson (Abstract No. 63)
9:30 A. M.—"The Electrolysis of Dibasic Acids and Their Mono-Acid Esters in Nonaqueous Solution" by W. E. Garrison, Jr., A. M. Hartley, Sherlock Swann, Jr., and C. S. Marvel (Abstract No. 64)
10:00 A.M.—"Electrolysis of Organic Solvents, I. Electrolysis of Simple Amides" by D. E. Couch and Abner Brenner (Abstract No. 65)
10:30 A.M.—"Studies of Germanium Electrodes in a Ziegler Electrolyte" by R. J. Flannery, D. Trivich, and J. E. Thomas, Jr. (Abstract No. 66)
11:00 A.M.—"Electrolysis in Anhydrous Ether" by I. E. Gillet (Abstract No. 67)
11:30 A.M.—"Electrolyte for Deposition at −60°C" by H. B. Maier (Abstract No. 68)
12:15 P.M.—Electrodeposition Division Luncheon and Business Meeting in the Yellow Room, Deshler Hilton Hotel.

Electrodeposition (cont'd)
Wednesday, October 21, 1959
Electro- and Chemical-Polishing
with D. G. Foulke presiding

(YELLOW ROOM, Deshler Hilton Hotel)

2:00 P.M.—"Electropolishing in Chloride Solutions" by E. B. Saubestre (Abstract No. 69)
2:30 P.M.—"Anodic Films on Copper in Phosphoric Acid" by J. H. Bartlett and F. H. Giles, Jr. (Abstract No. 70)
3:00 P.M.—"Polishing Regions for Copper in Phosphoric Acid" by J. H. Bartlett and F. H. Giles, Jr. (Abstract No. 71)
3:20 P.M.—"Application of Electropolishing and Chemical Polishing to Heat Sink Material" by P. T. Woodberry (Abstract No. 72)

3:50 P.M.—"Electropolishing in Cyanide Electrolytes" by F. Sullivan and E. H. Newton (Abstract No. 73)
4:20 P.M.—"Some Electrode Processes on Copper Anodes in Orthophosphoric Acid Solution" by K. F. Lorking (Abstract No. 74)

Electrodeposition (cont'd)
Thursday, October 22, 1959
General Session
with D. R. Turner presiding

(YELLOW ROOM, Deshler Hilton Hotel)

9:00 A.M.—"Electrical Migration in the Absence of Electrolysis" by Abner Brenner (Abstract No. 75)
9:30 A.M.—"Relationship between Brightness and Structure in Electroplated Nickel" by Rolf Weil and R. A. Paquin (Abstract No. 76)
10:00 A.M.—"Measurement of Elastic Modulus and Internal Friction in Electrodeposits" by H. J. Read and A. H. Graham (Abstract No. 77)
10:30 A.M.—"Electrodeposition of Amorphous Selenium" by A. K. Graham, H. L. Pinkerton, and H. J. Boyd (Abstract No. 78)
11:00 A.M.—"Zinc Plating of Carbonitrided Steel" by H. T. Francis and L. R. Kohan (Abstract No. 79)

Electrodeposition (cont'd)
Thursday, October 22, 1959
General Session
with Fielding Ogburn presiding
(YELLOW ROOM, Deshler Hilton Hotel)

2:00 P.M.—"Adherence of Thick Chromium Deposits as Affected by Surface Preparation" by H. Chessin and C. L. Alderuccio (Abstract No. 80)
2:30 P.M.—"Deposition of New Chromium-Iron Alloy Plate of Banded Structure" by L. D. McGraw, J. A. Gurklis, C. L. Faust, and J. E. Bride (Abstract No. 81)
3:00 P.M.—"Electrodeposition of Chromium from Low Valence Compounds, I. Investigation of Bath Types and Operating Conditions" by G. R. Sherwood, M. R. Holmes, and Finn Bergishagen (Abstract No. 82)
3:30 P.M.—"Studies of Hydrogen Embittlement by the Use of Deuterium" by G. B. Wood (Abstract No. 83)
4:00 P.M.—"The Source of the Nitrogen Impurity in Electrodeposited Chromium" by N. Ryan and E. J. Lumley (Abstract No. 84)

ELECTRONICS—SEMICONDUCTORS

Wednesday, October 21, 1959
Elemental Semiconductors, Bulk Properties
with H. H. Kedesdy presiding
(BALLROOM, Deshler Hilton Hotel)

9:00 A.M.—"Determination and Identification of Impurities in Silicon from Low-Temperature Hall

Data" by R. T. Bate, I. R. Weingarten, and D. J. Shombert (Abstract No. 85)

9:15 A.M.—"Use of Hall Measurements in Evaluating Polycrystalline Silicon" by P. J. Olshefski, D. J. Shombert, and I. R. Weingarten (Abstract No. 86)

9:35 A.M.—"Radio Frequency Carrier and Capacitive Coupling Procedures for Resistivity and Lifetime Measurements on Silicon" by I. R. Weingarten and M. Rothberg (Abstract No. 87)

9:55 A.M.—"Steady-State Diffusion of Charge Carriers in High-Purity Single-Crystal Silicon" by Milton Green and I. N. Greenberg . (Abstract No. 88)

10:20 A.M.—"Lifetime in Germanium as a Function of Gas Adsorption" by M. S. Fink and M. J. Katz (Abstract No. 89)

10:40 A.M.—Fifteen-minute intermission

10:55 A.M.—"Analysis of de Haas-van Alphen Type Oscillations in Graphite" by D. E. Soule (Abstract No. 90)

11:20 A.M.—"Some Optical and Electrical Properties of Crystalline Boron" by G. K. Gaulé, J. T. Breslin, J. R. Pastore, C. G. Pochop, and R. A. Shuttleworth (Abstract No. 91)

11:40 A.M.—"Diffusion of Injected Carriers in Silicon" by C. H. Champness (Abstract No. 92)

12:00 M.—Recent News Papers. Titles and short abstracts will be available at the meeting.

Electronics—Semiconductors (cont'd)
Wednesday, October 21, 1959
Semiconducting Compounds
with G. A. Silvey presiding
(BALLROOM, Deshler Hilton Hotel)

2:00 P.M.—"Preparation and Electrical Properties of Alloyed P-N Junctions of InSb" by C. A. Lee and George Kaminsky (Abstract No. 93)

2:20 P.M.—"Properties of Ternary Alloys in the System In-Sb-Te" by A. J. Rosenberg (Abstract No. 94)

2:40 P.M.—"Determination and Properties of Some Elements in GaAs" by E. Arnold, J. Black, and S. Weissberger (Abstract No. 95)

3:00 P.M.—"Mechanism of Preferential Etching of A^{III} B^{V} Compounds" by H. C. Gatos, M. C. Lavine, and M. J. Button (Abstract No. 96)

3:20 P.M.—Fifteen-minute intermission

3:35 P.M.—"Preparation and Electrical Properties of Mercury Selenide" by T. C. Harman (Abstract No. 97)

3:55 P.M.—"Mechanical and Infrared Excitation of Luminescence in CdS Crystals" by D. M. Warschauer and D. C. Reynolds (Abstract No. 98)

4:15 P.M.—"Properties of CdSb and CdSb-ZnSb Alloys" by A. J. Strauss (Abstract No. 99)

4:35 P.M.—"Physical Chemistry of the II-V Semiconductors CdSb and $CdAs_2$" by V. J. Lyons, V. J. Silvestri, and G. A. Silvey (Abstract No. 100)

4:50 P.M.—"Optical and Electrical Properties of CdSb and $CdAs_2$" by W. J. Turner, A. S. Fischler, and W. E. Reese (Abstract No. 101)

5:05 P.M.—"Phase Diagram for the Pseudo-Binary System CdTe-In_2Te_3" by Lars Thomassen and D. R. Mason (Abstract No. 102)

Electronics—Semiconductors (cont'd)
Thursday, October 22, 1959
Process Technology
with A. W. Postlethwaite presiding

(BALLROOM, Deshler Hilton Hotel)

9:00 A.M.—"Purification of Arsenic" by J. M. Whelan, J. D. Struthers, and J. A. Ditzenberger (Abstract No. 103)

9:20 A.M.—"Preparation of High-Purity Gallium" by Pierre de la Bretèque (Abstract No. 104)

9:45 A.M.—"Impurity Distribution of Various Doping Techniques in Floating Zone Crystal Growing" by A. L. MacDonald and E. Y. Wang (Abstract No. 105)

10:05 A.M.—"Vibratory Polishing and Lapping of Semiconductor Materials" by J. E. Cline and S. J. Solomon (Abstract No. 106)

10:25 A.M.—"Chemical Etching of Silicon, III. A Temperature Study in the Acid System" by Harry Robbins and Bertram Schwartz (Abstract No. 107)

10:40 A.M.—Fifteen-minute intermission

10:55 A.M.—"A Low Resistance Ohmic Contact for Silicon Semiconductor Devices" by S. L. Matlow and E. L. Ralph (Abstract No. 108)

11:15 A.M.—"A Chemical Treatment for Rendering Silicon-Doped Aluminum Solderable" by S. L. Matlow and H. J. Gould (Abstract No. 109)

11:35 A.M.—"Depletion Layer Calculations for the Design of Diffused Semiconductor Devices" by H. Lawrence and R. M. Warner, Jr. (Abstract No. 110)

11:55 A.M.—"Development of Damage-Nucleated Structures in Silicon by Highly Preferential Etching" by P. R. Pennington and J. D. Turner (Abstract No. 111)

12:05 P.M.—"Silicon Transistors and Device Fabrication Using Damage-Nucleated Etched Structures" by W. P. Waters and P. Walker (Abstract No. 112)

There is no Abstract No. 113

Electronics—Semiconductors (cont'd)
Thursday, October 22, 1959
Surface Phenomena, Diffusion
with E. I. Doucette presiding

(BALLROOM, Deshler Hilton Hotel)

2:00 P.M.—"A New Approach to Semiconductor Surface Reactivity" by G. A. Wolff (Abstract No. 114)

2:25 P.M.—"Theory of Surface Conductance in Semiconductors" by D. R. Frankl (Abstract No. 115)
2:45 P.M.—"P-Layers on Vacuum Heated Silicon" by J. T. Law, T. M. Buck, and F. G. Allen (Abstract No. 116)
3:05 P.M.—"Surface Channel Studies on Silicon P-N Junctions" by Raymond Solomon and P. O. Johnson (Abstract No. 117)
3:25 P.M.—Fifteen-minute intermission
3:40 P.M.—"Diffusion in Silicon Carbide" by Hung-Chi Chang and L. F. Wallace (Abstract No. 118)
4:00 P.M.—"Surface Concentration of Indium in Germanium from Gaseous Diffusion" by K. M. Busen, E. L. Meeks, and G. A. Shirn (Abstract No. 119)
4:20 P.M.—"Capillary Alloying: An Improved Alloying Method" by K. M. Busen, J. J. Casey, Kurt Lehovec, and A. C. Webb (Abstract No. 120)
4:45 P.M.—"Impurity Redistribution and Junction Formation in Silicon by Thermal Oxidation" by M. M. Atalla and E. Tannenbaum (Abstract No. 121)
5:05 P.M.—"Metallurgical Analysis of the Bulk Diffusion Phenomenon in Silicon Power Transistors" by P. X. Flaherty (Abstract No. 122)
5:25 P.M.—"Diffusion of Phosphorus into Silicon through an Oxide Layer" by D. Kahng and M. O. Thurston (Abstract No. 122A)

ELECTRO-ORGANIC

Monday, October 19, 1959
with Garrett Thiessen presiding

(YELLOW ROOM, Deshler Hilton Hotel)

9:30 A.M.—"Application of Constant Potential Electrolysis to the Process for Electrolytic Formation of Hydrazobenzene in Alkaline Emulsion. Mechanism for the Processes at Soluble and Insoluble Cathodes" by Taro Sekine, Tadashi Seki, and Kiichiro Sugino (Abstract No. 123)
10:30 A.M.—"Preparation of Crossed-Pinacols by Cathodic Reduction" by M. J. Allen and W. Pierson (Abstract No. 124)
11:00 A.M.—"Kolbe Decomposition Potentials by Condenser and Ballistic Galvanometer" by G. W. Thiessen and F. F. Rawlings (Abstract No. 125)
11:30 A.M.—"Electrolytic Formation of Oximes" by Tadao Hayashi and C. L. Wilson (Abstract No. 126)

Electro-Organic (cont'd)
Monday, October 19, 1959
with C. L. Wilson presiding

(YELLOW ROOM, Deshler Hilton Hotel)

2.00 P.M.—"The Stereo Selective Electrochemical Reduction of Nonconjugated Steroidal Ketones and Alpha Ketols" by Peter Kabasakalian, Alvin Basch, James McGlotten, and Milton Yudis (Abstract No. 127)

2:30 P.M.—"Electrochemical Research in India and Japan" by M. J. Allen (Abstract No. 128)

Electro-Organic (cont'd)
Tuesday, October 20, 1959
with R. A. Day, Jr., presiding
(YELLOW ROOM, Deshler Hilton Hotel)

9:00 A.M.—"Polarographic Reduction of Some Acetylenic Linkages" by A. H. Gropp and J. C. Ramsey, Jr. (Abstract No. 129)
9:30 A.M.—"Polarographic Reductions of Benzyl Halides" by L. W. Marple, L. E. I. Hummelstedt, and L. B. Rogers (Abstract No. 130)
10:00 A.M.—"Polarography in Formamide, I. Some Transition Metal Ions, Zinc and Cadmium Ions" by G. H. Brown and Hsiao-shu-Hsiung (Abstract No. 131)
10:30 A.M.—"Polarographic Studies in Dimethylformamide, VI. Reduction of Polyhalogenated Hydrocarbons" by Stanley Wawzonek and R. C. Duty (Abstract No. 132)
11:00 A.M.—"Millicoulometric Determination of Polarographic n-Values" by Thos. De Vries and J. L. Kroon (Abstract No. 133)
12:15 P.M.—Electro-Organic Luncheon and Business Meeting in the Yellow Room, Deshler Hilton Hotel.

Electro-Organic (cont'd)
Tuesday, October 20, 1959
with Stanley Wawzonek presiding
(YELLOW ROOM, Deshler Hilton Hotel)

2:00 P.M.—"Polarographic Reduction of Some Diketones" by R. L. Flurry and R. A. Day, Jr. (Abstract No. 134)
2:30 P.M.—"Polarographic Reduction of Nonconjugated Steroidal Ketones" by Peter Kabasakalian and James McGlotten (Abstract No. 135)
3:00 P.M.—"Electrolytic Reduction of Some Alkylnitrosamines" by G. C. Whitnack, R. D. Weaver, and H. W. Kruse (Abstract No. 136)
3:30 P.M.—"Rotating Cathode-Type Cell for the Preparation of Benzidine" by K. S. Udupa, G. S. Subramanian, and H. V. K. Udupa (Abstract No. 136A)
5:00 P.M.—Palladium Medal Address by A. N. Frumkin in the Ballroom, Deshler Hilton Hotel.

ELECTROTHERMICS AND METALLURGY

Monday, October 19, 1959
Refractory Coatings
with J. M. Blocher, Jr., presiding
(PARLOR B, Neil House)

9:30 A.M.—"Refractory Ceramic Coatings Today and Tomorrow" by F. D. Shaw (Abstract No. 137)
10:00 A.M.—"The Adhesion of Vitreous Oxide Coatings to Metals" by B. W. King and W. H. Duckworth (Abstract No. 138)

10:30 A.M.—"A Liquid Phase Coating for Refractory Metals" by G. D. Oxx, Jr., and L. F. Coffin, Jr. (Abstract No. 139)

11:00 A.M.—"Testing Procedures for Evaluation of Coated Materials" J. L. Harp and J. M. Allen (Abstract No. 140)

Electrothermics and Metallurgy (cont'd)
Monday, October 19, 1959
Refractory Coatings (Cont'd)
with David Roller presiding

(PARLOR B, Neil House)

2:00 P.M.—"Problems of Applying Metallic Coatings to Nonmetallic Bases" by S. W. Bradstreet (Abstract No. 141)

2:30 P.M.—"Coating Uranium Dioxide Powders with Metallic Films" by J. H. Oxley, J. F. Hannah, J. M. Blocher, Jr., and I. E. Campbell (Abstract No. 142)

3:00 P. M.—"Silicon Carbide Coatings on Graphite Paper" by J. G. Kemp (Abstract No. 143)

3:30 P.M.—"Metal-Ceramic Coatings for Protection of Graphite" by W. L. Aves (Abstract No. 144)

Electrothermics and Metallurgy (cont'd)
Tuesday, October 20, 1959
Refractory Coatings (cont'd)
with M. A. Steinberg presiding

(PARLOR B, Neil House)

9:30 A.M.—"Inorganic Coatings for Leading Edges of Boost-Glide Vehicles" by H. A. Pearl (Abstract No. 145)

10:00 A.M.—"Coatings for Structural Aircraft Materials" by D. H. Leeds (Abstract No. 146)

10:30 A.M.—"Refractory Coatings for Ramjet and Supersonic Aircraft Applications" by S. Sklarew (Abstract No. 147)

11:00 A.M.—"Protective Coatings for Medium Temperature Ranges" by A. R. Stetson (Abstract No. 148)

Electrothermics and Metallurgy (cont'd)
Tuesday, October 20, 1959
Less Common Metals
with A. C. Loonam presiding

(PARLOR B, Neil House)

2:00 P.M.—"Reactions in the Niobium-Hydrogen System" by W. M. Albrecht, W. D. Goode, and M. W. Mallett (Abstract No. 149)

2:30 P.M.—"A Study of the Iodide Niobium Process" by R. F. Rolsten (Abstract No. 150)

3:00 P.M.—"The Behavior of Rhenium in Electron Tube Environments" by G. B. Gaines, C. T. Sims, and R. I. Jaffee (Abstract No. 151)

5:00 P.M.—Palladium Medal Address by A. N. Frumkin in the Ballroom, Deshler Hilton Hotel.

Abstracts

In the Index to Authors, the Abstract Number is the number listed in the right-hand column.

BATTERY

Abstract No. 1

Study of the Recuperation Reaction in the Leclanché Dry Cell

M. P. Korver, Research Labs., National Carbon Co., Division of Union Carbide Corp., Cleveland, Ohio, **R. S. Johnson,** Riegel Textile Corp., Ware Shoals, S. C., and **N. C. Cahoon,** Edgewater Development Lab., National Carbon Co., Cleveland, Ohio

The cathodic reaction in the Leclanché dry cell has been described previously as consisting of two steps. The first is the reduction of Mn^{IV} to Mn^{II} and the second is the reaction of Mn^{II} produced in the first step with unreacted Mn^{IV} to form an insoluble Mn^{III} by-product. Two reactions can occur producing MnOOOH and $ZnO \cdot Mn_2O_3$, manganite and hetaerolite, respectively. The latter reactions, termed recuperation reactions, have been subjected to analysis and rate studies to determine the effects of factors such as pH, concentration, and temperature. The basic concepts of heterogeneous chemical kinetics have been applied and a simple mathematical equation was found which was applicable to all the data. These findings support the cathodic reaction mechanism theory presented previously.

Abstract No. 2

A Method of Increasing the Service Life of Leclanché Dry Cells

H. J. DeWane, National Bureau of Standards, Washington 25, D. C.

A simple, inexpensive rectifying system for "charging or rejuvenating" dry cells has been investigated. The circuit consists of a filament transformer, a Ge diode in parallel with a resistor, a current limiting lamp, and three batteries in series. D-size cells were charged for a number of cycles after completion of standard ASA performance tests, and the electrical output or increased service was calculated. Some cells were also cycled continuously. The latter procedure results in a greater gain in service.

Abstract No. 3

A Method for Evaluating Battery Grade Manganese Dioxide

Friedrich Kornfeil, Energy Conversion Research Branch, U. S. Army Signal Research and Development Lab., Fort Monmouth, N. J.

A method is described for evaluating manganese dioxide as a cathode material. The principle of this method is to discharge a mixture of MnO_2 and acetylene black at a constant effective current density in a simply constructed test cell. The circuitry used eliminates the potential drop due to the internal resistance of the test cell, thereby adding substantially to the simplicity of the preparation of the sample for the test. Data are presented which show the correlation of the results of this technique with those of industrial high drain (16-2/3 ohm) capacity tests of dry cells containing the same oxides.

Abstract No. 4

An Improved Iodide Method for the Analysis of Manganese Dioxide

F. J. Krivanek and **N. C. Cahoon,** Development Div., National Carbon Co., P. O. Box 6056, Cleveland 1, Ohio

The accurate determination of the available oxygen content of manganese dioxide samples is of considerable importance to the battery industry, since this value is often used to indicate the suitability of the sample for use. A study of a recently proposed method based on the liberation of iodine in an acid solution has resulted in significant improvements in accuracy and reliability. The revised method gives excellent results with synthetic samples. However, the lack of satisfactory results using the improved method with ore samples suggests that interfering substances are present.

Abstract No. 5

Impedance of Leclanché Cells

R. J. Brodd and **H. J. DeWane,** National Bureau of Standards, Washington 25, D. C.

Cells with various MnO_2: acetylene black ratios were constructed and discharged on standard tests. The impedance of the cells was measured over the frequency range 50-50 kc/s by a substitution method. Results are interpreted from an analysis of Cole-Cole impedance plots.

Abstract No. 6

The Overpotentials of Some Oxide Electrodes and the Discharge Mechanism

W. C. Vosburgh, J. H. DeLap, and **H. B. Mark, Jr.,** Dept. of Chemistry, Duke University, Durham, N. C.

Discharge of electrodeposited MnO_2 electrodes for short periods at *p*H 8, with time for reattainment of equilibrium, shows that the potential of partially discharged MnO_2 is a linear function of the amount of lower oxide formed, for part of the discharge. With this relationship, some previous data on the overpotential

of MnO_2 in acid electrolytes are interpreted more simply than before, and a similar relationship and mechanism can be applied to PbO_2 and other oxide electrodes.

Abstract No. 7

Gravity Field Effects on Zinc Anode Discharge in Alkaline Media

M. Eisenberg and **H. F. Bauman,** Electrochemistry Lab., Lockheed Missiles and Space Division, Sunnyvale, Calif.

This study was undertaken as a follow-up to theoretical predictions relating to the behavior of batteries with soluble anodes in gravity-free fields, such as are encountered in space flight. Since weightlessness conditions cannot be obtained under ordinary circumstances in the laboratory, an indirect experimental approach was selected. Concentration gradients and interfacial concentration of dissolved zinc were determined at 3 different orientations to the direction of the gravitational field at the zinc anode surface by means of slow boundary sampling methods. Through a correlation of these results with mass transfer theory of free convection, predictions for the behavior of zinc anodes in gravity-free fields are made.

Abstract No. 8

Magnesium Primary Cells with Perchlorate Electrolytes

G. S. Lozier and **C. K. Morehouse,** RCA Semiconductor and Materials Div., Radio Corp. of America, Somerville, N. J.

The desirable properties of an aqueous perchlorate electrolyte in magnesium primary cells are: high anode efficiency, low tendency to complex, and stability in presence of strong oxidizing agents. These properties have led to the development of magnesium primary cells with HgO, AgO, NiO, CuO, and MnO_2 cathodes. Magnesium dry cells containing CuO and MnO_2 cathodes have a favorable shelf life and give greater watt-hour capacities on a weight and volume basis than Leclanché dry cells.

Abstract No. 9

A Synchronized Square Wave Generator and Switching Unit for Electrochemical Studies

N. C. Cahoon and **H. F. Schaefer,** National Carbon Battery Development Labs., National Carbon Research Labs., Cleveland, Ohio

An interrupter for producing a repetitive square wave pulse of direct current is combined with a synchronized switching relay for electrochemical studies. This unit provides a means of obtaining semi-instantaneous readings of either the open or closed cell voltage or electrode potential of an electrochemical system

when in operation. The change in shape of the voltage-time pulse, displayed on an oscilloscope screen, shows the extent to which the square wave is modified by the electrode system and provides a useful tool for studying electrodes.

Abstract No. 10

Sine Wave Pulse Current Tester for Batteries

K. Kordesch, Research Labs., National Carbon Co., 12900 Snow Rd., Parma, Ohio, and **A. Marko,** Wright Air Development Center, Fairborn, Ohio

The separation of the polarization and resistance component of the voltage drop occurring in a battery under load conditions is measured with a 60-cycle, a-c operated instrument which is portable, allows direct readings without an oscilloscope, and is especially suited for development and production control tasks. Results of measurements on Leclanché cells, alkaline primary and secondary batteries, lead acid batteries, and fuel cells are presented. The relation to cell performance is discussed.

Abstract No. 11

The Silver/Silver-Oxide Electrode System

B. D. Cahan, J. B. Ockerman, R. Amlie, and **Paul Rüetschi,** Research Center, The Electric Storage Battery Co., Yardley, Pa.

Constant current anodization of silver in KOH was studied using a high-speed recorder with an electrometer input. Growth of anodic films was measured with a superimposed square-wave technique whereby interface resistance and capacitance were followed continuously. The mechanisms for the conversion of Ag to Ag_2O, of Ag_2O to AgO, and of the evolution of oxygen are discussed in detail. Decay of oxygen overvoltage on AgO has been studied and correlated with BET areas. Discharge and self-discharge reactions and efficiency of discharge of each species have been studied similarly.

Abstract No. 12

Oxides on the Silver Electrode

C. P. Wales and **Jeanne Burbank,** U. S. Naval Research Lab., Washington 25, D. C.

Silver electrodes were studied by potential and x-ray diffraction patterns. Silver was charged anodically in 35% KOH by either constant current or constant potential and discharged at a low or high rate. Attempts were made to form anodically an oxide higher than AgO. The anodic products of silver in 2N H_2SO_4 were determined. The basis for the theory that silver oxides are formed by introducing oxygen into the octants of the face-centered cubic silver lattice was examined.

Abstract No. 13

The Stability and Solubility of AgO in Alkaline Solutions

T. P. Dirkse and **Brandon Wiers,** Dept. of Chemistry, Calvin College, Grand Rapids, Mich.

AgO is stable in boiling water and decomposes but very little at room temperature in strongly alkaline solutions. The presence of Ag_2O does not affect this stability, but the presence of unoxidized silver does increase the rate of decomposition. AgO decomposes at temperatures above 100°C. When AgO dissolves in strongly alkaline solutions the dissolved form is primarily AgO and $AgO(OH)_2^-$. The standard free energy of formation of the latter is —85,380 cal/mole.

Abstract No. 14

The Oxidation of the Silver Electrode in Alkaline Solutions

T. P. Dirkse, Dept. of Chemistry, Calvin College, Grand Rapids 6, Mich.

The formation of Ag_2O and AgO was studied by means of constant current and constant potential processes. The efficiency of these oxide formations was determined by measuring the gain in weight of Ag electrodes under various conditions of charge, and the extent of the polarization of these processes was measured by using an interrupted current technique.

Abstract No. 15

The Effect of Cycling on the Silver Electrode

T. P. Dirkse and **J. De Vries,** Dept. of Chemistry, Calvin College, Grand Rapids, Mich.

Sintered silver electrodes have been cycled at various current rates and temperatures. An attempt is made to determine whether this treatment alters the capacity or physical characteristics of the silver electrode.

Abstract No. 16

Factors Affecting the Photogalvanic Behavior of Silver-Silver Halide Electrodes

S. Zaromb, M. Lasser, and **F. Kalhammer**, Research Dept., Philco Corp., Philadelphia, Pa.

The quantum yields of photochemically formed Ag available for electrolytic discharge in Ag-AgCl and Ag-AgBr cells depended mainly on method of electrode preparation, electrolyte composition, and light wavelength, but did not vary with the intensity of illumination for average solar radiation. Direct recombination of the photochemical reaction products and corrosion of Ag by an oxidant in the electrolyte are the main yield limiting factors.

Abstract No. 17

Silver Halide Photogalvanic Batteries

S. Zaromb, M. Lasser, and **L. Bockstie,** Research Dept., Philco Corp., Philadelphia, Pa.

Batteries rechargeable by the photolysis of silver halides are obtained by effecting spontaneous separation and storage of the photochemical reaction products utilizing selectively permeable membrane arrangements. Initial and continuous quantum yields vary with electrolyte compositions and electrode and membrane compositions and structure. The results are interpreted in terms of reaction rates, carrier mobilities, local energy potentials, and rectifying interfaces.

Abstract No. 18

Burgess Mercury Cells

T. H. Loverude, Burgess Battery Co., Division of Servel, Inc., Freeport, Ill.

A brief description is made of the general operating principles of the mercury cell. Several unique features of the Burgess mercury cells are outlined, and a description of the Burgess Hg-3 cell is made showing how they have controlled venting, simplified electrical contact between the cathode and the cathode can, and other construction features.

Abstract No. 19

Investigation of the Electrochemical Characteristics of Organic Compounds, V. Heterocyclic Nitro Compounds

R. Glicksman and **C. K. Morehouse,** RCA Semiconductor and Materials Div., Radio Corp. of America, Somerville, N. J.

A study of the electrochemical characteristics of nitropyridine and other heterocyclic nitro compounds shows that the cathode potential of these compounds during current flow is dependent on the aromaticity of the compound, as well as the type and position of substituent groups on the aromatic ring. The effect of these factors on the cathode potential is interpreted in terms of the electron density distribution in the molecule. Performance characteristics of magnesium dry cells containing 2,5-dinitrofuran and various nitropyridine compounds as cathodes are also presented.

Abstract No. 20

Investigation of the Electrochemical Characteristics of Organic Compounds, VI. Aromatic Hydroxy, Aromatic Amine, and Aminophenol Compounds

R. Glicksman, RCA Semiconductor and Materials Div., Radio Corp. of America, Somerville, N. J.

A study of the electrochemical characteristics of aromatic hydroxy, aromatic amine, and aminophenol

compounds in basic electrolyte shows that the anode potential of these compounds is dependent on the aromaticity of the compound, as well as the type and position of substituted groups on the aromatic ring. The effect of these factors on the anode potential is interpreted in terms of the electron density distribution in the molecule. The use of these compounds as anode materials in primary cells is also considered

Abstract No. 21

The Principal Source of αPbO_2 in the Positive Plate Material of the Lead-Acid Battery

V. H. Dodson, Electric Auto-Lite Co., Toledo Ohio

An examination of positive plate material (*a*) formed from positive pastes of various densities (cube weights), (*b*) formed in aqueous sulfuric acid solutions of various specific gravities, and (*c*) at various stages of development indicates that the major source of α-PbO_2 in the positive material is unsulfated lead oxide. A method for estimating α- and β-PbO_2 in a mixture of the two is also described.

Abstract No. 22

The PbO_2 Electrode

R. T. Angstadt, B. D. Cahan, and **P. Rüetschi,** Research Center, The Electric Storage Battery Co., Yardley, Pa.

Electrode potentials of α-PbO_2 and β-PbO_2 were measured in solutions of sulfuric acid and perchloric acid with controlled concentration of Pb^{++} ion, as a function of *p*H and temperature. The double layer capacity of PbO_2 electrodes has been studied over a wide range of potentials under steady-state and transient conditions with the use of a square wave technique. The range of thermal stability and transition points of α-PbO_2 and β-PbO_2 have been determined.

Abstract No. 23

The Microscopy of the Effect of Environmental Changes on the Forming Process

A. C. Simon and **E. L. Jones,** U. S. Naval Research Lab., Washington 25, D. C.

The effect of varying temperature, acid concentration, and current density were studied in relation to the microstructure produced. To determine the effective limits of the process, temperature was varied from 4° to 80°C, current density from 0.23 to 2.3 amp/dm^2 and acid from 1.050 to 1.450 sp gr. While the actual efficiency of the forming process was not determined under these varying environmental conditions, it was found that the microstructure and extent of formation varied widely.

Abstract No. 24

The Microscopy of the Forming Process in the Positive and Negative Plate

A. C. Simon and **E. L. Jones,** U. S. Naval Research Lab., Washington 25, D. C.

To determine the suitability of microscopy for such a purpose an exploratory study was made of the microscopic changes in structure that occur during the forming process. Samples taken at spaced intervals during the forming operation were prepared for microscopic examination by means of plastic impregnation. The examination more or less confirmed the generally held views on the subject, but emphasized the need for further investigation as to the effect of environment.

Abstract No. 25

Internal Resistance of Lead-Acid Cells; Submarine Battery Size

J. J. Lander and **E. E. Nelson,** Naval Research Lab., Washington, D. C.

Lead-acid cells of submarine battery size were discharged through resistance-inductance circuits. The data are analyzed to show no contribution to voltage loss other than *IR* losses in the cells during the initial voltage transient; consequently, a method is provided for the unequivocal determination of the internal resistance. Circuit geometry is an important factor determining the types of discharge curves which can be obtained.

Abstract No. 26

Nickel-Cadmium Cell Electrodes

A. J. Salkind and **P. F. Bruins,** Research Center, The Electric Storage Battery Co., Yardley, Pa.

Voltage decays of electrodes in nickel-cadmium cells were measured in cells stored at temperatures between −18° and 52°C. The heat of reaction of the cell was calculated from the slope of a plot of E/T vs. the logarithm of the vapor pressure of water and was found to be −62 kcal/g mole. The solid reaction products of the cell were studied by x-ray techniques.

Abstract No. 27

Investigations on the Reaction Mechanism of the Nickel-Cadmium Cell

S. Uno Falk, Svenska Ackumulator AB Jungner, Stockholm, Sweden

X-ray diffraction patterns from electrodes submerged in electrolyte have been obtained during charge and discharge by means of a special test cell, and the

composition of the active materials has been determined. The question whether CdO or $Cd(OH)_2$ is formed during discharge has been subject to special attention. The low potential reaction of the positive plate has been studied. The emf and the dE/dT have been determined between −40° and +50°C at various states of charge.

Abstract No. 28

Sealed Nickel-Cadmium Cells Containing Palladium

H. N. Seiger, AVCO, RAD, Wilmington, Mass.

An investigation of the charge and discharge characteristics of cells containing palladium as a H_2-O_2 recombination catalyst was carried out. Preliminary measurements of the internal pressure indicate the catalytic coefficient for Pd:Ni is 1.4. The charge and discharge characteristics of the cells are unaffected by the pressure of the palladium catalyst.

CORROSION

Abstract No. 29

The Attack of Copper-Gold, Silver-Gold, Copper-Nickel, and Silver-Copper Alloys by Sulfur at Elevated Temperatures

B. D. Lichter (present address: Air Force Cambridge Research Center, Bedford, Mass.) and **Carl Wagner** (present address: Max-Planck Institut fur Physikalische Chemie, Gottingen, Germany), Dept. of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

Cu-Au alloys involving 10 and 33 mole % Au and Ag-Au alloys involving 12 mole % Au are attacked by liquid or gaseous sulfur above 400°C very rapidly with formation of a composite scale consisting of an outer homogeneous sulfide layer and an inner two-phase layer involving Au-rich alloy and sulfide in accord with theoretical considerations regarding the instability of a plane alloy-sulfide interface. The tendency to form a rugged alloy-sulfide interface decreases with higher Au contents especially at lower temperatures, and the rate of attack by sulfur decreases accordingly. Ag-Cu alloys involving 40 mole % Cu are also rapidly sulfidized at 400°C and yield a single sulfide layer of uniform thickness. Cu-Ni alloys sulfidized at 400°C yield a scale consisting of an outer layer which is supposedly digenite $Cu_{1.8}S$, and an inner layer which is supposedly a solid solution of Cu_2S in nickel sulfide.

Abstract No. 30

Anodic Oxidation of High-Purity Aluminum in Water

W. E. Tragert and **J. R. Rairden,** Research Lab., General Electric Co., P. O. Box 1088, Schenectady, N.Y.

The effect of applying an anodic potential during the oxidation of high-purity aluminum in double-distilled

water has been studied with particular reference to the influence of an electric field on the crystal structure and composition of the oxide formed. The films can be characterized by nearly cubic close-packing of anions and by a temperature dependence of composition. Results are reported at several anodizing temperatures and over a range of anodizing currents.

Abstract No. 31

The High-Temperature Aqueous Corrosion Resistance of the Uranium-5% Zirconium-1½% Niobium Alloy*

J. E. Draley, Sherman Greenberg, and **W. E. Ruther,** Argonne National Laboratory, Lemont, Ill.

Uranium-5% zirconium-1½% niobium shows good corrosion resistance, as gamma quenched, in water to about 315°C. Optimum heat treatment results in a rate of about 6 mg/cm²/day at 260°C. Moderate aging, e.g., 400°C for 2 hr, results in reduced corrosion rates in the initial stages of corrosion, but corrosion resistance can be destroyed by over-aging. The alloy is also sensitive to hydrogen content. Ultimate failure is believed due to absorption of corrosion product hydrogen.

* Work performed under auspices of USAEC.

Abstract No. 32

Evidence for a Logarithmic Oxidation Process for Stainless Steel in Aqueous Systems

Milton Stern, Metals Research Labs., Union Carbide Metals Co., Division of Union Carbide Corp., Niagara Falls, N.Y.

Upon immersion of stainless steel into ferrous-ferris solution, the potential gradually approaches the reversible potential of the solution. During this period, an oxidation process occurs, the rate of which decreases with time. The observed kinetics are consistent with equations which describe ion current through thin oxides as a function of the field across the oxide and are similar to kinetics reported for anodic oxidation of metals which form thin amorphous oxides. Therefore, it is believed that evidence has been obtained for growth of a similar type of oxide on passive stainless steel.

Abstract No. 33

Passivating Inhibitors

A. C. Makrides and **Milton Stern,** Technology Dept., Union Carbide Metals Co., P. O. Box 580, Niagara Falls, N.Y.

Results of an experimental study of the mechanism of passivation of Ti, Ni, and various steels by oxidizing inhibitors are presented. In particular, experiments were designed to differentiate between adsorption and reduction of inhibitor. It is suggested that for the cases examined inhibitor adsorption, if any, is of minor im-

portance. Passivity is obtained only if the reduction rate of the oxidizing inhibitor exceeds a certain critical value characteristic of the metal. For a number of systems, the reduction rate is limited only by the rate of transport of inhibitor to the metal-solution interface. Thus, whether passivity or acceleration (of dissolution) is observed depends on mass transport rates which are, in turn, dependent on hydrodynamic flow conditions.

Abstract No. 34

Dissolution of Metals in Aqueous Acid Solutions

A. C. Makrides, Technology Dept., Union Carbide Metals Co., P. O. Box 580, Niagara Falls, N.Y.

A previously proposed interpretation of potential-current density curves obtained in dissolution of metals in aqueous acid solutions is examined in detail. It is shown that the dissolution rate-electrode potential curve for iron, nickel, and chromium-iron alloys, in solutions containing other redox couples besides H^+/H_2, closely approximates the anodic polarization curve of the metal. The bearing of these findings on the mechanism of passivating inhibitors proposed by Stern is discussed briefly.

Abstract No. 34A

Potential Behavior of a Uranium Electrode as a Function of Partial Pressure of Oxygen*

J. W. Ward and **J. T. Waber,** University of California, Los Alamos Scientific Lab., P. O. Box 1663, Los Alamos, N. Mex.

The potential of a uranium electrode coated with oxide was measured as a function of the partial pressure of oxygen. Although the equilibria involved are established slowly, the potential values were reproducible. The behavior with only traces of oxygen present was considered. An explanation is offered for the observed behavior.

* Work done under the auspices of the A.E.C.

Abstract No. 35

An Electrochemical Study of Passive Films on Fe-Cr Alloys

D. Caplan, A. Harvey, and **M. Cohen,** Division of Applied Chemistry, National Research Council, Ottawa, Canada

An attempt to deduce the nature of films left on Fe-Cr alloys by various surface preparations was made using electrochemical measurements. Electron microscopy and diffraction and parallel experiments with Cr and Fe were carried out for purposes of clarification. Cathodic reduction after certain preparations such as

electropolishing yields an anomalous transient high negative potential suggesting an outer film of high hydrogen overvoltage which is slowly cathodically reduced to expose a surface of lower hydrogen overvoltage. With highly passive films it was found necessary to follow the potential during etching in order to remove the passive film without overetching.

Abstract No. 36

Effect of Surface Preparation on Air-formed Films on Iron

C. D. Stockbridge, P. B. Sewell, and **M. Cohen,** National Research Council of Canada, Ottawa, Canada

Surfaces of pure iron single and polycrystals have been prepared by electropolishing and electropolishing with subsequent etching or cathodic reduction. The nature of the surfaces has been investigated electrochemically and by electron diffraction. Electropolishing in the perchloric-acetic bath leaves a thin oxide on the surface which changes its thickness and structure with time of exposure to air. This film is different from that formed after either cathodic reduction or light acid treatment of the electropolished surface. In all cases the oxides are cubic and are either Fe_3O_4 or γFe_2O_3 depending on the orientation and surface treatment.

Abstract No. 37

Nitric-Hydrofluoric Acid Pickling of Zirconium Alloys

M. A. DeCrescente (present address: CANEL, Middletown, Conn.) **P. F. Santoro, A. S. Powell,** and **R. H. Gale,** Materials Development Lab., Nuclear Div., Combustion Engineering, Inc., Windsor, Conn.

The dissolution of Zircaloy-2 has been studied in the temperature range of 60°-100°F in unagitated and agitated hydrofluoric-nitric acid solutions. The dissolution rate was found to increase with HF concentration and agitation. The energy of activation for the Zircaloy-2 pickling process compares very well to the value for the unalloyed zirconium pickling process, 3.3 kcal/mole. The energy of activation does not depend on agitation.

Abstract No. 38

An Investigation of Chemical Variables Affecting the Formation of Films on Copper in Aqueous Solutions

W. H. Davenport, V. F. Nole, and **W. D. Robertson** (present address: Yale University, New Haven, Conn.)
Chase Brass and Copper Co., Waterbury, Conn.

The growth of copper oxide films in aqueous solutions has been studied by an electrometric method. Quantitative data have been obtained on the growth of films as a function of the solution variables, anion type and concentration, pH, and oxygen concentration. In air-stirred chloride solutions, film thickness increases

with increasing chloride concentration, passes through a maximum in solutions approximately 0.01 molar, and then decreases. A maximum is also obtained for pH dependence in the range pH 5.9-9.0. Cuprous oxide is formed below about pH 10.5, and cupric oxide predominates at higher values.

Abstract No. 39

Dissolution of Single Crystals of Copper in Aqueous Ethylenediamine

L. H. Jenkins, Solid State Div., Oak Ridge National Lab., Oak Ridge, Tenn.

Single crystal plates of 99.999% copper were exposed to aqueous solutions of ethylenediamine saturated with air. Rates of dissolution of low index faces at fixed stirring rates between 7° and 60°C were determined and the activation energy for the process was calculated. Observations of the metal surfaces with optical and electron microscopes at different stages of the reaction revealed the effects of crystal orientations and imperfections on facet formation and pitting of the surfaces.

Abstract No. 40

The Electrochemical Behavior and Passivity of Titanium

Milton Stern and **Herman Wissenberg,** Metals Research Labs., Union Carbide Metals Co., Division of Union Carbide Corp., Niagara Falls, N.Y.

In the active condition, corrosion of titanium is controlled by activation polarization. The metal presents a surface with a low exchange current for a variety of electrochemical reactions. Low exchange currents are not conducive to establishment of passivity. However, this is overcome by unusually active critical potentials for passivity. This accounts for the ease with which titanium is passivated by many substances which are only slightly oxidizing. It also explains the ease with which the metal can be protected anodically by galvanic coupling to other metals and alloys. Anodic deposition of thick titanium oxide films is observed under some experimental conditions.

Abstract No. 41

The Influence of Noble Metal Alloy Additions on the Electrochemical and Corrosion Behavior of Titanium

Milton Stern and **Herman Wissenberg,** Metals Research Labs., Union Carbide Metals Co., Division of Union Carbide Corp., Niagara Falls, N.Y.

Alloying of titanium with small amounts of noble metals markedly improves its corrosion resistance in reducing-type acids without any impairment of its high resistance to oxidizing media. The mixed potentials of titanium noble metal alloys are more noble than the critical potential for passivity of titanium in the same

environment. Passivity is observed in hydrogen-saturated systems without the presence of a redox system more oxidizing than the hydrogen electrode. The mixed potential of titanium noble metal alloys is much more sensitive to traces of oxidizing agents than unalloyed metal.

CORROSION—ELECTRONICS

Abstract No. 42

Introduction to the Physics and Chemistry of Surfaces

W. H. Brattain, Bell Telephone Labs., Murray Hill, N.J.

The fundamental concepts of the physics and chemistry of surfaces will be considered starting with Langmuir's work on thermionic emission and adsorption on metal surfaces and ending with the simple surface or phase boundary that occurs at a p-n junction in a semiconductor. The semiconductor surface seems to be the better model for most catalytic surfaces. Future progress depends on measuring both physical and chemical changes on the same surface at the same time.

Abstract No. 43

Metal Surfaces

H. J. Juretschke, Dept. of Physics, Polytechnic Institute of Brooklyn, 333 Jay St., Brooklyn 1, N.Y.

The paper reviews the concepts which have been found useful in the discussion of various properties of metal surfaces. It includes a summary of theoretical work done in this area, predictions made by theory, and a discussion of experimental results and what they tell about the relation of the theory to actual surfaces.

Abstract No. 44

Semiconductor Surfaces

Paul Handler, University of Illinois, Urbana, Ill.

A brief history of the research in semiconductor surface physics is presented. Emphasis is placed on the limits of present theory and the importance of knowing the composition and structure of the surface of interest. The feasibility of new experimental approaches to the study of surfaces, such as nuclear magnetic resonance and quadropole resonance, is discussed. A review of recent developments in understanding the energy level diagram of the cleaned germanium surface is reviewed.

Abstract No. 45

Clean Surfaces

H. E. Farnsworth, Dept. of Physics, Brown University, Providence, R.I.

The method of obtaining atomically clean surfaces of refractory materials by prolonged heating at high

temperatures in ultra-high vacua cannot be applied to most materials. Two alternative methods have been used in ultra-high vacua: (*a*) cleaving of crystals, and (*b*) argon-ion bombardment followed by annealing. The first method is limited to materials and conditions which permit cleaving. The second method has been applied more generally and has been found effective for a compound with low melting point as well as for more refractory elements and compounds.

Abstract No. 46

The Role of Imperfections in Surface Reactions

N. Cabrera, University of Virginia, Charlottesville, Va.

(No abstract received)

Abstract No. 47

Damaged Surface Layers—Semiconductors

T. M. Buck, Bell Telephone Labs., Inc., Murray Hill, N.J.

Preparation of semiconductor surfaces usually involves cutting and lapping or polishing. The damage produced by abrasion of this sort causes marked changes in surface recombination velocity, field effect mobility, conductivity, etching rate, and other properties. The type of abrasive material and the manner of treatment strongly influence the depth of the damaged layer. Estimates of these depths by various methods of detection will be presented along with discussion of the nature of the damage.

Abstract No. 48

Effect of Imperfections on Dissolution

J. J. Gilman, Research Lab., General Electric Co., Schenectady, N.Y.

At high rates of dissolution, the process is controlled by the rate of diffusion in the solvent, but surface imperfections play an important and often dominant role when dissolution is slow. Adsorbed species, dislocations, and point-defect clusters are the imperfections involved. They interact in highly specific ways. This is revealed by the sensitivity of the shapes of etch-pits to adsorbed species in the solvent and impurities in the solid for the case of LiF crystals; to whether an In or an Sb atom is at the center of a dislocation in the case of InSb crystals; and to optical activity in the solvent in the case of calcite crystals.

Abstract No. 49

Metal and Semiconductor Electrode Processes

H. Gerischer, Max Planck Institute, Stuttgart, Germany

The rate of surface reactions as electrode processes combined with transfer of electrically charged particles between the neighboring phases depends on the

strength of electrical fields in the interface and on electron reactivity. The different order of magnitude of mobile electrons in a metal and in a semiconductor as well as the different distribution of states cause important differences in the distribution of charges near the interface and in the surface reactivity of electrons. The influence of these phenomena on electrode reaction kinetics with ion transfer or electron transfer at the interface will be discussed and compared with some examples.

Abstract No. 50

Experimental Techniques for the Study of Semiconductor Electrodes

J. F. Dewald, Bell Telephone Labs., Murray Hill, N.J.

Experimental techniques for the study of semiconductor electrodes are reviewed, and the theory involved in the treatment of the resulting data is discussed for several specific cases. A new transient technique, which is particularly useful with large-energy-gap semiconductors, is described.

Abstract No. 51

Electrode-Solution Interfaces

H. J. Engell, Max Planck Institut fur Eisenforschung, Düsseldorf, Germany

The differences of the distribution of charges at the surfaces of a metal electrode and a semiconductor electrode are considered. Using a simplified model the densities of the charge carriers in a semiconductor electrode can be calculated. The result of the calculation is in agreement with measurements of the differential capacity of germanium electrodes. The influence of the current passing through the electrode surface and of the formation of traps is considered.

Abstract No. 52

Electrolytic Attack of Metals

Paul Lacombe, Case Institute of Technology, Cleveland, Ohio

Electrolytic etching of metals produces various results: intergranular attack, attack of crystalline surfaces which is orientation dependent, formation of etch pits, and anodic oxide films. The behavior of a metal or alloy depends on composition, temperature of the electrolyte, and above all on the electrode potential which varies with the metal. The method involving variation of the potential permits the establishment of the best conditions for the specific attack of each phase in an alloy. Applications to Al, Fe, stainless steel, Ti, Zr, U, and their alloys will be discussed.

Abstract No. 53

Electrolytic Etching of Semiconductors

D. R. Turner, Bell Telephone Labs., Inc.. Murray Hill, N.J.

Anodic reactions at semiconductor electrodes require electron holes. If the hole concentration in the semiconductor is relatively low, as in low resistivity n-type Ge or Si, the available holes in the surface region are used up at low current densities, and the etch rate is slow. The anodic current under these conditions can be increased by providing additional holes at the surface. Holes produced as a result of illuminating the semiconductor give uniform electrolytic etching on n-type semiconductors. Germanium is electrolytically etched in several electrolytes while silicon can only be dissolved anodically in fluoride solutions.

Abstract No. 54

The Etching of Metals and Semiconductors

J. W. Faust, Jr., Research Labs., Westinghouse Electric Corp., Pittsburgh, Pa.

Etching may be defined as the controlled removal of surface atoms. Various physical and chemical changes that can be used for etching are reviewed briefly. Studies on those aspects of dissolution of metals and semiconductors that relate to etching are discussed in more detail. Similarities and differences in the etching of metals and semiconductors are taken up, using specific systems for illustration.

Abstract No. 55

Kinetics of Dissolution Processes

Norman Hackerman, Dept. of Chemistry, University of Texas, Austin 12, Texas

The kinetics of dissolution of solids is governed in general by transport phenomena, or by whatever process is required to disrupt the primary aggregating forces in the solid, or by some combination of both. In principle the steps can be separated experimentally and studied individually. For processes which are controlled by transport the problems, both theoretical and experimental, are those of fluid dynamics and of the diffusion layer in nonturbulent systems. For processes in which the rate depends on bond disruption the principal step may be one of solvation or it may involve an oxidation-reduction system. The latter is predominant in most cases involving metals, and then it becomes necessary to determine which, if either, of the electron transfer processes is limiting. Most attention is given to problems in the last area.

Abstract No. 56

Electrochemistry of Dissolution Processes

J. V. Petrocelli, Research Labs., The International Nickel Co., Inc., Bayonne, N.J.

The basic electrochemical concepts and ideas underlying the phenomena of metal dissolution are reviewed.

The emphasis is on the electrochemistry of metallic corrosion in aqueous solutions. The role of oxidation potentials as a measure of the "driving force" is discussed and the energetic factors which determine the relative electrode potential are described. It is shown that a consideration of electrochemical kinetics, in terms of current-voltage characteristics, allows an electrochemical classification of metals and leads to the modern views of the electrochemical mechanism of corrosion and passivity.

Abstract No. 57

Dissolution of Metals

C. V. King, Dept. of Chemistry, New York University, New York, N.Y.

In solutions of reagents which form soluble products and in regions of nonpassivating electrical potentials, metals often dissolve at rates controlled by convective-diffusive transport of reagent. The characteristics of such rates and some empirical and theoretical aspects of the "diffusion layer" will be discussed. Conditions under which transport control is replaced by other types of control will be discussed and examples will be presented. Adsorption of a reaction product may inhibit dissolution, or complete surface coverage by a necessary intermediate may limit the dissolution rate, for example.

Abstract No. 58

Dissolution of Semiconductors*

H. C. Gatos, Lincoln Lab., Massachusetts Institute of Technology, Lexington, Mass.

There are many similarities between semiconductors and metals regarding their dissolution kinetics, passivity, and electrochemistry in aqueous solutions. These similarities will be discussed particularly with reference to germanium. On the other hand, semiconductivity and structure are responsible for certain unique features associated with the dissolution behavior of semiconductors. Specific adsorption of ions, for instance, plays a prominent role in the dissolution kinetics of germanium. In addition to the behavior of elementary semiconductors that of certain intermetallic compound semiconductors will be discussed.

* The work reported in this paper was performed by Lincoln Laboratory, a center for research operated by Massachusetts Institute of Technology with the joint support of the U.S. Army, Navy, and Air Force.

Abstract No. 59

Adsorption and Chemisorption

M. Boudart, Princeton University, Princeton, N.J.

(No abstract received)

Abstract No. 60

Chemisorption and Catalysis

R. P. Eischens, Texaco Research Center, Beacon, N.Y.

The most direct evidence concerning chemisorbed molecules is obtained from their infrared spectra. These spectra show the structure of the chemisorbed molecules, effect of interactions between these molecules, and effect of modifying the adsorbent metal by other components. Infrared results show that the chemistry of the surface metal atoms is similar to that of metal atoms in coordination compounds. This tends to restore catalysis to the domain of the chemist and suggests that factors inherent in the physical structure of metals, such as lattice defects or surface geometry, are not essential to the basic catalytic process.

Abstract No. 61

On the Mechanism of the Oxidation of Metals

K. Hauffe, Farbwerk Hoechst AG, Frankfurt (Main), Germany

After a short introduction on the relationship between diffusion process and field transport phenomena in tarnishing layers on metals and alloys, the mechanism of oxidation of iron is discussed. Epitaxy plays an important role on the gradient of the concentration of lattice defects and, therefore, on the validity of the parabolic rate law. Classical examples of metal oxidation with a parabolic rate law are presented and the various reasons for the deviation observed are elucidated on the systems iron in CO/CO_2 and Cu_2O in O_2. In addition, the oxidation of alloys with interrupted oxide-metal interfaces is treated. Finally, attention is focussed on the difficulties in explaining the low temperature-oxidation mechanism.

Abstract No. 62

The Influence of Crystal Orientation on Oxidation of Metals

A. T. Gwathmey and **K. R. Lawless,** Dept. of Chemistry, University of Virginia, Charlottesville, Va.

The influence of crystal orientation on the rate of formation and on the epitaxy of oxide films formed on large metal crystals will be discussed. Past studies will be reviewed, new results will be presented, and emphasis will be placed on important questions which must yet be answered in order to understand oxidation. Primary consideration will be given to crystals of copper.

ELECTRODEPOSITION

Abstract No. 63

Anodic Methoxylation

K. E. Kolb, and **T. W. Lippincott,** Ohio State University, Columbus, Ohio, and **C. L. Wilson,** Cranberry Lake, Sloatsburg, N.Y.

Electrolysis of sodium methoxide in methanol at a graphite or platinum anode in an undivided cell gives

formaldehyde, sodium formate, and sodium methyl carbonate. If furan or thiophene is also present, two methoxyl groups are added to the molecules to give 2,5-dimethoxy-2,5-dihydrofuran (or thiophene). The yield is as high as 50%. If, instead of direct, alternating current is used, no methoxylation reaction is observed until the frequency is lowered to three cycles or less per second. This suggests that an intermediate radical is formed on the anode with an unexpectedly long lifetime of about 20 sec.

Abstract No. 64

The Electrolysis of Dibasic Acids and Their Mono-Acid Esters in Nonaqueous Solution

W. E. Garrison, Jr., A. M. Hartley, Sherlock Swann, Jr., and **C. S. Marvel,** Dept. of Chemistry and Chemical Engineering, University of Illinois, Urbana, Ill.

The electrolysis of a series of ammonium salts of aliphatic dicarboxylic acids, in methanol, has been carried out. Polymers were formed at the anode from adipic, azelaic, sebacic, and hexadecane-1,16-dicarboxylic acids. These polymers could be separated into soluble and insoluble fractions. The soluble fractions were found to be of low molecular weight and linear, with some ester linkages. The dimethyl ester of dotriacontane-1,32-dicarboxylic acid was synthesized in 57% yield by electrolysis of the ammonium salt of monomethyl hexadecane-1,16-dicarboxylate.

Abstract No. 65

Electrolysis of Organic Solvents, I. Electrolysis of Simple Amides

D. E. Couch and **Abner Brenner,** National Bureau of Standards, Washington 25, D.C.

Except for water and ammonia, the electrolysis of few solvents has been investigated. A general project on the electrolysis of organic solvents has been initiated to study the products and the development of a mechanism of the electrode reactions. This first report deals with the electrolysis of several simple amides.

Crystalline compounds have been produced by the electrolysis of N,N'dimethylformamide, N-methylformamide and N,N'dimethylacetamide with bright platinum electrodes. These materials were formed by the electrolysis of anhydrous solvent and by the electrolysis of the solvent containing 5-10% by volume of 1M H_2SO_4. However, if water is present in large amounts, 50% by volume, the yield of product is reduced and impurities which make separation of the product difficult are formed. Little or no gas is formed at the anode but at the cathode hydrogen is produced with 95-100% efficiency.

Abstract No. 66

Studies of Germanium Electrodes in a Ziegler Electrolyte

R. J. Flannery (present address: Research Labs., Standard Oil Co. (Indiana), 2500 New York Ave.,

Whiting, Ind.) **D. Trivich,** and **J. E. Thomas, Jr.,** Wayne State University, Detroit, Mich.

The behavior of n- and p-type germanium electrodes, particularly under anodic conditions, was studied in a Ziegler electrolyte composed of triethyl aluminum and sodium fluoride. D-C anodic and A-C current-voltage curves indicate current saturation effects in n-type but not in p-type germanium. Germanium does not dissolve anodically, and the anode products are characteristic of a reaction of ethylide ions with holes. The cathode product, aluminum, forms a rectifying junction on n-Ge or p-Si.

Abstract No. 67

Electrolysis in Anhydrous Ether

I. E. Gillet, Chef de Travaux d'Electrochimie et de Chimie Organique Appliquee, Universite de Liege, 2, rue A. Stevart, Liege, Belgium

Electrolyses have been carried out on anhydrous ethyl ether made conductive by dissolution of inorganic salts or Grignard reagents ($AlCl_3$ or $AlCl_3 + LiCl$ or $MgBr_2$ or $RMgBr$). Polarization curves have been determined and products analyzed. Results and conclusions are presented, the main conclusion being that ether itself reacts easily at both electrodes and is sometimes the only reacting substance, the dissolved salt being used only to conduct the current in solution.

Abstract No. 68

Electrolyte for Deposition at —60°C

H. B. Maier, Bergen Labs. Inc., Clinton, N.J.

An electrolyte which is liquid and conductive at —60°C consists of ethanol, water, sodium sulfate, nickel sulfate, and additional inorganic and organic materials. The salts are dissolved in the water, and a large amount of ethanol (approximately 80% by volume) is added to form an electrolyte solution having a low freezing point. Conduction at —60°C, after running overnight, is 1.5 ma at 13 v for electrodes 1 $in.^2$ in area, ⅞ in. apart. The anode consisted of a strip of nickel, the cathode of a strip of copper. During removal of the electrolysis cell from the freezing unit, no gassing was observed. A small amount of black powder was seen on the cathode. Further tests are being conducted to obtain a uniform metallic coating at —60°C.

Abstract No. 69

Electropolishing in Chloride Solutions

E. B. Saubestre, Enthone, Inc., A Subsidiary of American Smelting and Refining Co., 442 Elm St., New Haven, Conn.

Solution formulas and operating conditions are presented which permit electropolishing of a wide number

of metals and alloys in solutions containing chlorides. These solutions resemble the perchloric acid type of electropolishing solution in many respects. Outstanding feature of these solutions is the very wide range of current densities over which electropolishing occurs. These solutions are most useful for treating small parts.

Abstract No. 70

Anodic Films on Copper in Phosphoric Acid

J. H. Bartlett, University of Illinois, Dept. of Physics, Urbana, Ill., and **F. H. Giles, Jr.,** University of South Carolina, Columbia, S.C.

If initially a copper anode in phosphoric acid is in the steady state and if the current is then interrupted, break curves of the variation of voltage with time may be recorded. At the lower voltages, such curves decay in a manner resembling an exponential, and the convection layer disappears at the same time. If the voltage is just above the point corresponding to the knee of the current-voltage curve, there is superimposed on the above break curve a similar one with a much smaller time constant. This is what would be observed if concentration gradients in a much thinner layer were being ironed out. This time constant increases with voltage, indicating that the thickness does, also.

Abstract No. 71

Polishing Regions for Copper in Phosphoric Acid

J. H. Bartlett, University of Illinios, Dept. of Physics, Urbana, Ill., and **F. H. Giles, Jr.,** University of South Carolina, Columbia, S.C.

By microscopic observations of a vertical copper anode during electrolysis in phosphoric acid, the dependence of electropolishing on voltage and current density has been recorded. In 50% H_3PO_4, two regions of applied voltage were found in which polishing occurred: a lower region just at and above the knee of the steady-state current-voltage curves (between 0.1 and 0.26 v with respect to 0.1*N* calomel) and an upper region which extended from a steady-state voltage of approximately 0.8 v with respect to 0.1 calomel up into the region of oxygen evolution.

The sensitivity of the polishing surface to electrical and mechanical changes in each of these regions of polishing has been observed, as well as the variation with acid concentration of the extent of the polishing regions themselves. These data are discussed in terms of our knowledge of the Cu-H_3PO_4 anodic system, and the significance to the theory of the mechanism of electropolishing is considered.

Abstract No. 72

Application of Electropolishing and Chemical Polishing to Heat Sink Material

P. T. Woodberry, Avco, RAD, 201 Lowell St., Wilmington, Mass.

Electropolishing and chemical polishing techniques were used to prevent the occurrence of defective zones

of copper-copper interfaces in the electrodeposition of thick copper. Electropolishing could be substituted for grinding and machining of nodules prior to subsequent layer deposition. Copper was polished, electropolished, or chemically polished before deposition of thick nickel; the effect of surface preparation and type of copper on the elevated temperature properties of this system are discussed.

Abstract No. 73

Electropolishing in Cyanide Electrolytes

F. Sullivan and **E. H. Newton,** Arthur D. Little, Inc., Cambridge, Mass.

The theory and practice of electropolishing silver, sterling silver, gold alloys, and brass in cyanide electrolytes are described. Commercial electropolishing of silver plate, pilot plant experience in electropolishing sterling silver, and laboratory investigations into gold alloys and brass electropolishing are covered. Compositional and operational variables in each process are reviewed.

Abstract No. 74

Some Electrode Processes on Copper Anodes in Orthophosphoric Acid Solution

K. F. Lorking, Aeronautical Research Labs., Box 4331, G. O. P., Melbourne, Australia

Theories on the structure of the electrical double layer formed at polarizable anode surfaces and on electrode processes at reacting anodes are used to indicate the processes to be expected on copper anodes in orthophosphoric acid solutions. A description and discussion are given of experiments showing the influence of increasing anode potential on surface finish and film formation on copper anodes in orthophosphoric acid. Theories of the mechanism of electropolishing are discussed and it is shown that modifications of these theories are indicated by experimental observations.

Abstract No. 75

Electrical Migration in the Absence of Electrolysis

Abner Brenner, National Bureau of Standards, Washington 25, D.C.

Electrical migration of ions has always been observed in experiments involving electrolysis. J. W. Richards believed that electrical migration was a secondary effect of current flow and was due to stresses set up by the electrolytic decomposition at the electrodes. He considered that the mechanism of the flow of current in electrolytes was the same as that in metals.

An experiment was devised for examining electrical migration in the absence of electrolysis. By means of a magnetic field and a liquid commutator, a direct

current was induced into a closed circuit consisting of electrolyte. No electrolysis was involved. Electrical migration was determined by the movement of Erythrosin B into a capillary. The experiment showed that electrical migration occurred in the absence of electrolysis and was qualitatively of about the same magnitude as that observed in the presence of electrolysis.

Abstract No. 76

Relationship between Brightness and Structure in Electroplated Nickel

Rolf Weil and **R. A. Paquin,** Dept. of Metallurgy, Stevens Institute of Technology, Hoboken, N.J.

The as-plated surface microstructure of deposits of varying brightnesses was examined by electron microscopy. A linear relationship was found between the fraction of the surface area with roughness less than 0.15 μ and the logarithm of the quantity of light reflected. No direct relationship between the degree of preferred orientation and brightness was found, but the fiber axis is related to the structural type and the addition agents in the bath.

Abstract No. 77

Measurement of Elastic Modulus and Internal Friction in Electrodeposits

H. J. Read and **A. H. Graham,** Dept. of Metallurgy, The Pennsylvania State University, University Park, Pa.

Experimental techniques and calculations for the determination of elastic modulus and internal friction from sonic measurements on thin-walled tubes prepared by electrodeposition are described. A method of making the tubes with the necessary precision is presented. Data on the effects of some plating variables are given, but special attention is devoted to relationships between the structures of the deposits and the elastic properties under study.

Abstract No. 78

Electrodeposition of Amorphous Selenium

A. K. Graham, H. L. Pinkerton, and **H. J. Boyd,** Graham, Savage and Associates, Inc., Jenkintown, Pa.

Amorphous selenium coatings have been electroplated in thicknesses up to (and thicker coatings can be prepared) 30 μ (1.2 mils) on bright nickel cathodes. Data are given for studies leading to the selection of the optimum bath and operating conditions for the formation of reproducible, uniform, and adherent deposits. It is shown that the plating baths employed and the amorphous deposits obtained are extremely sensitive to a variety of conditions, thus emphasizing the need for further study of the electrodeposition of selenium in the amorphous form.

Abstract No. 79

Zinc Plating of Carbonitrided Steel*

H. T. Francis and **L. R. Kohan,** Armour Research Foundation, 10 West 35th St., Chicago 16, Ill.

A fundamental study was made of the difficulty sometimes encountered in barrel zinc plating of carbonitrided steel parts. A very successful laboratory technique was devised for simulation of barrel plating in a still plating bath. Results suggest that the difficulty in achieving zinc coverage on the hardened parts is caused by a "plate and dissolve" mechanism.

* This work was supported by the Shakeproof Division of Illinois Tool Works.

Abstract No. 80

Adherence of Thick Chromium Deposits as Affected by Surface Preparation

H. Chessin and **C. L. Alderuccio,** Van der Horst Corp. of America, Olean, N.Y.

Photomicrographs show that honing, a mild and slow-speed operation as compared to either grinding or machining, may cause extensive damage to the surface of cast iron. Chromium electrodeposited on these damaged surfaces will exhibit poor adherence not because the adherence of the chromium to the iron is poor but because the surface layer of iron is weak.

Abstract No. 81

Deposition of New Chromium-Iron Alloy Plate of Banded Structure

L. D. McGraw, J. A. Gurklis, and **C. L. Faust,** Electrochemical Engineering Div., Battelle Memorial Institute, Columbus 1, Ohio, and **J. E. Bride,** Diamond Alkali Co., Cleveland, Ohio

A new bath composition and its operating conditions are described for electrodeposition of hard, bright, chromium-iron alloy plate having banded microstructure. A precipitation mechanism is proposed to account for the microstructure of the plate.

Abstract No. 82

Electrodeposition of Chromium from Low Valence Compounds, I. Investigation of Bath Types and Operating Conditions

G. R. Sherwood, M. R. Holmes, and **Finn Bergishagen,** Dept. of Chemistry, Wayne State University, Detroit 2, Mich.

Excellent quality chromium metal can be deposited at comparatively good efficiency from baths in which the chromium is chiefly trivalent. Presence of divalent chromium in such baths does not seem essential but does seem beneficial. A mixed bath, containing chiefly chloride-formate-fluoborate, is recommended rather than a bath containing one salt only.

Abstract No. 83

Studies of Hydrogen Embrittlement by the Use of Deuterium

G. B. Wood, National Bureau of Standards, Washington 25, D.C.

Fundamental studies dealing with the passage of the hydrogen isotopes through SAE 4130 steel are discussed. The rate of passage of hydrogen through steel, the amount of hydrogen causing embrittlement, and the resulting severity of embrittlement are correlated. The use of deuterium as the embrittling isotope permits a differentiation between hydrogen electrolytically introduced into steel and that introduced due to metallurgical procedures. Hydrogen embrittlement of steel accompanying cadmium and chromium plating and alkaline and acid electrolytic treatment in the presence and absence of poisons are discussed.

Abstract No. 84

The Source of the Nitrogen Impurity in Electrodeposited Chromium

N. Ryan and **E. J. Lumley,** Aeronautical Research Labs., Dept. of Supply, Box No. 4331, G. P. O., Melbourne, C. 1, Vic., Australia

High-purity chromium deposited from chromic acid electrolytes containing either sulfate or fluoride catalysts invariably contains small amounts of nitrogen as an undesirable impurity. The amount of this nitrogen impurity is shown to be increased by increased nitrate ion concentration in the electrolyte but not affected by dissolved atmospheric nitrogen or ammonium compounds. It is demonstrated that the nitrate impurity is decomposed at the cathode by reduction with hydrogen to form ammonia as the major product. Some of the atomic nitrogen which is possibly formed at a stage during this reduction is probably adsorbed continuously into the electrodeposited metal.

ELECTRONICS—SEMICONDUCTORS

Abstract No. 85

Determination and Identification of Impurities in Silicon from Low-Temperature Hall Data

R. T. Bate, Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio, **I. R. Weingarten,** and **D. J. Shombert,** Research Lab., Merck, Sharp and Dohme, Rahway, N.J.

Low-temperature measurements of the Hall effect have been used to determine donor and acceptor concentrations and in some cases to identify the major impurity in silicon. A simple apparatus for making these measurements with liquid helium is described.

Typical results are presented and correlated with mobility data at 80° or 55°K and lifetime data at room temperature. Interpretation of the Hall coefficient measurements is discussed.

Abstract No. 86

Use of Hall Measurements in Evaluating Polycrystalline Silicon

P. J. Olshefski, Electronic Chemicals Div., Chemical Div., Merck & Co., Inc., Danville Pa., **D. J. Shombert,** and **I. R. Weingarten,** Research Labs., Merck, Sharp and Dohme, Rahway, N.J.

Hall measurements at room temperature have been used to determine the average net carrier density in polycrystalline silicon. The method does not require cutting of samples and destruction of the rod. Current contacts are made to the ends of the rod with strips of metal foil; Hall contacts are made with two titanium blades which close on the rod. Hall measurements made this way on single-crystal zone-refined rods agree with measurements on samples cut from the rods in the conventional manner. Average carrier densities measured in the polycrystalline rods have been correlated with the resistivities of the same rods after 1, 2, 3. . . floating zone passes.

Abstract No. 87

Radio Frequency Carrier and Capacitive Coupling Procedures for Resistivity and Lifetime Measurements on Silicon

I. R. Weingarten and **M. Rothberg,** Research Labs., Merck, Sharp and Dohme, Rahway, N.J.

By the use of the rf carrier and capacitive coupling it has been possible to measure the resistance and lifetime of silicon rods without making direct ohmic contact to the sample. Even with contacts, the use of an rf carrier to observe lifetime by photoconduction decay eliminates the need for insuring solid ohmic contacts as is required with the d-c carrier. With the low-loss insulation between coupling capacitors and sample, an rf bridge will measure separately both the sample resistance between the coupling capacitors and the value of the coupling capacitance.

Abstract No. 88

Steady-State Diffusion of Charge Carriers in High-Purity Single-Crystal Silicon

Milton Green and **I. N. Greenberg,** U. S. Army Signal Research and Development Lab., Fort Monmouth, N.J.

Results of irradiating high-purity silicon (ca 1900 ohm cm p-type and 590 ohm cm n-type) with light near metal-to-silicon contact junctions (all found to be rectifying at room temperature) were investigated. An

expression is presented for the photocurrent at steady-state illumination (focused as a very narrow line parallel to the junction), one-dimensional geometry, and sample long compared to a diffusion length. In the preliminary experiments it was found that the diffusion phenomenon was masked by the photoeffect due to light scattered directly to the junction. Since the scattered light is most difficult to eliminate, it was minimized by using a broad line of light of variable breadth extending from the junction to a certain distance along the specimen. Expressions for the photocurrent under this condition and one-dimensional geometry are presented and compared with experiment.

Abstract No. 89

Lifetime in Germanium as a Function of Gas Adsorption

M. S. Fink and **M. J. Katz,** U. S. Army Signal Research and Development Lab., Fort Monmouth, N.J.

Changes of lifetime in germanium have been measured as a function of adsorption of various gases at different temperatures, pressures, and time. Data show an interesting dependence of the direction and magnitude of the changes on the nature of the adsorbate and its interaction with the adsorbing surface. Interpreted in terms of adsorption theory, the measurements imply a better understanding of the chemical origin of surface states and their effect on electronic properties.

Abstract No. 90

Analysis of de Haas-van Alphen Type Oscillations in Graphite

D. E. Soule, National Carbon Co., Research Labs., Cleveland, Ohio

Oscillations of the de Haas-van Alphen type have been observed in the Hall effect and magnetoresistance in graphite single crystals. They have been studied as a function of carrier type (majority electrons and holes), magnetic field orientation, temperature, and acceptor doping. Analysis of results has revealed the anisotropy and fine structures of the Fermi surface, effective masses, collision broadening, carrier concentrations, Fermi energies, and electronic band overlap.

Abstract No. 91

Some Optical and Electrical Properties of Crystalline Boron

G. K. Gaulé, J. T. Breslin, J. R. Pastore, C. G. Pochop, and **R. A. Shuttleworth,** U.S. Army Signal Research and Development Lab., Fort Monmouth, N.J.

Optical transmission measurements have been made on boron crystals obtained from a melt and from a vapor phase. Essential differences in the absorption

coefficient vs. wave-length curves were observed. Most samples were chemically rather impure, p-type, of high resistivity, and had a high Seebeck coefficient. Some material, however, showed a more "metallic" character. Contacts were nearly ohmic in all cases; photoconductivity was always small. Potential technical uses of some optical and electrical properties are discussed.

Abstract No. 92

Diffusion of Injected Carriers in Silicon

C. H. Champness, Northern Electric Labs., 1401 St. Patrick St., Montreal, Que., Canada

The field-free diffusion of minority carriers injected at a point into silicon has been studied. The time from injection to the maximum due to the arriving carriers has been measured for various emitter collector distances. Results do not agree with the conventional treatment of diffusion and life time. There are indications that, due to heavy injection of carriers, there is enhanced recombination resulting in an effective life time which decreases as the emitter point is approached.

Abstract No. 93

Preparation and Electrical Properties of Alloyed P-N Junctions of InSb

C. A. Lee and **George Kaminsky,** Bell Telephone Labs., Inc., Murray Hill, N.J.

A study of p-n junction characteristics at liquid nitrogen temperatures has been made on junctions prepared by alloying cadmium to n-type InSb. Reports of junctions in InSb have been published, but, apart from sensitivity of detection of infrared radiation, no other junction characteristics were presented. Our investigations indicate that cadmium seems to be unique among the Group II and VI elements in that p-n junctions, exhibiting good rectifying properties, can be made with conventional alloying techniques.

Abstract No. 94

Properties of Ternary Alloys in the System In-Sb-Te*

A. J. Rosenberg, Lincoln Lab., Massachusetts Institute of Technology, Lexington, Mass.

Four known semiconducting binary compounds (InSb, InTe, In_2Te_3, Sb_2Te_3) exist in the system, In-Sb-Te. Together with elemental Te and (amorphous) Sb, they comprise a group of six semiconducting phases with fundamentally different crystal sym-

* The work reported in this paper was performed by Lincoln Laboratory, a center for research operated by Massachusetts Institute of Technology, with the joint support of the U.S. Army, Navy, and Air Force.

metry, chemical bonding, and electronic properties. A study of the general phase relationships and electronic characteristics of ternary compositions of In, Sb, and Te has been undertaken, and the preliminary data are discussed.

Abstract No. 95

Determination and Properties of Some Elements in GaAs

E. Arnold, J. Black, and **S. Weissberger,** Sylvania Electric Products Inc., Bayside, N.Y.

The concentration of residual impurities and of added donor and acceptor elements in zone melted GaAs was determined by spectroscopic, spectrophotometric, and neutron activation analyses. Approximate values of the effective segregation coefficients and solubility limits of elements from Groups II, IV, VI of the periodic table were determined from chemical analysis and metallographic examination of doped ingots. The net donor or acceptor concentrations, as determined from chemical analysis, were compared with the free carrier concentrations as determined from Hall measurements on single crystals. It was found that the two concentrations do not always agree. The agreement is discussed for a number of elements in relation to their concentration and position in the periodic table, with reference to such factors as dislocation density and heat treatment.

Abstract No. 96

Mechanism of Preferential Etching of $A^{III}B^{V}$ Compounds*

H. C. Gatos, M. C. Lavine, and **M. J. Button,** Lincoln Lab., Massachusetts Institute of Technology, Lexington, Mass.

Etching of intermetallic compounds with the zinc-blende structure has been studied in several aqueous media as a function of temperature and orientation. Examination of the etching characteristics of the{III} and {III} planes indicates that no dislocation etch-pits are revealed in one of these planes in a large number of aqueous solutions, although dislocation etch-pits are consistently revealed in the other. By introducing small amounts of various elements into the zinc-blende lattice, etch-pits are revealed in both planes. A model is proposed for explaining this phenomenon based on lattice vacancies and edge dislocations.

* The work reported in this paper was performed by Lincoln Laboratory, a center for research operated by Massachusetts Institute of Technology with the joint support of the U.S. Army, Navy, and Air Force.

Abstract No. 97

Preparation and Electrical Properties of Mercury Selenide*

T. C. Harman, Lincoln Lab., Massachusetts Institute of Technology, Lexington, Mass.

Large single-crystal ingots of mercury selenide have been prepared by the Bridgman technique. The technique used for the reaction and crystallization of the compound is described. At 77°K, most pure material possessed a carrier concentration of $2 \times 10^{17}/cm^3$ and an electron Hall mobility of 63,000 $cm^2/v\text{-}sec$. Information on the HgSe-CdSe alloys and results of selective doping experiments are also presented.

* The work reported in this paper was performed by Lincoln Laboratory, a center for research operated by Massachusetts Institute of Technology with the joint support of the U.S. Army, Navy, and Air Force.

Abstract No. 98

Mechanical and Infrared Excitation of Luminescence in CdS Crystals

D. M. Warschauer and **D. C. Reynolds,** Wright Air Development Center, Wright Air Force Base, Ohio

Mechanically excited edge and impurity emission in thermally or optically stimulated cadmium sulfide will be discussed in terms of recent band models and a necessary set of impurity levels. The potential applications of this effect in computer storage devices, infrared detectors, etc., will be covered. Unlike the polarization of edge emission and the red luminescence of silver, the orientation of the electric vector of red emission from copper in cadmium sulfide is parallel to the c-axis.

Abstract No. 99

Properties of CdSb and CdSb-ZnSb Alloys*

A. J. Strauss, Lincoln Lab., Massachusetts Institute of Technology, Lexington, Mass.

The variation in thermoelectric power of CdSb samples prepared under different conditions of crystallization is described. The thermoelectric power depends strongly on whether CdSb is formed by direct crystallization or by crystallization of metastable Cd_3Sb_2 and subsequent solid-state reaction of the latter compound with Sb. Thermal analysis data on the phase diagram of the CdSb-ZnSb pseudo-binary system, together with x-ray and thermoelectric data on the alloys, are also presented.

* The work reported in this paper was performed by Lincoln Laboratory, a center for research operated by Massachusetts Institute of Technology with the joint support of the U.S. Army, Navy, and Air Force.

Abstract No. 100

Physical Chemistry of the II-V Semiconductors CdSb and $CdAs_2$

V. J. Lyons, V. J. Silvestri, and **G. A. Silvey,** Research Center, International Business Machines Corp., Poughkeepsie, N.Y.

The thermal dissociation of CdSb and $CdAs_2$ may be represented by the following equations:

$$3CdAs_2 \rightleftharpoons Cd_3As_2 + As_4 \qquad [1]$$

$$CdSb \rightleftharpoons Cd + Sb \qquad [2]$$

A study of the reactions under equilibrium conditions has provided measurements of the dissociation pressures for the compounds. Crystal pulling of the compounds was carried out in sealed quartz tubes wherein magnetic suspension of the seed and crystal was employed. Several methods of purification were used during preparation of the compounds. A comparison of results indicates that the highest degree of purification was obtained through segregation of impurities during crystal pulling. Experiments are described relating to the effect of annealing on monocrystalline $CdAs_2$ under equilibrium conditions.

Abstract No. 101

Optical and Electrical Properties of CdSb and $CdAs_2$

W. J. Turner, A. S. Fischler, and **W. E. Reese,** Research Center, International Business Machines Corp., Poughkeepsie, N.Y.

CdSb has an optical gap of 0.51 ev and transmission to 32 μ. The net electron concentration in the single crystal CdSb studied was approximately 10^{16} cm^{-3} and had room temperature Hall mobility of 250 cm^2/v-sec.

$CdAs_2$ is tetragonal with a gap of 1.11 ev and optimum transmission in the c-direction from the edge to 35 μ. Pulled $CdAs_2$ crystals showed net electron concentrations of 10^{15} cm^{-3} to 10^{17} cm^{-3}. Hall mobilities over 300 cm^2/v-sec have been observed in the 10^{15} cm^{-3} material. The conductivity of the c-direction $CdAs_2$ is a factor of four larger than the a-direction over the temperature range of 77°-300°K.

Abstract No. 102

Phase Diagram for the Pseudo-Binary System CdTe-In_2Te_3*

Lars Thomassen and **D. R. Mason,** Dept. of Chemical & Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

The phase diagram for pseudo-binary system CdTe-In_2Te_3 has been investigated by correlating information

* Supported by Project MICHIGAN and the National Science Foundation.

from differential thermal analysis measurements, microscopic studies, and x-ray powder patterns. In checking the terminal points for this diagram, the melting point for CdTe was found to be 1098° ± 2°C, which represents a considerable deviation from the previously reported value of 1045° C. No congruently melting compounds form along this particular line in the ternary system Cd-In-Te.

Abstract No. 103

Purification of Arsenic

J. M. Whelan, J. D. Struthers, and **J. A. Ditzenberger,** Bell Telephone Labs., Inc., Murray Hill, N.J.

Small concentrations of sulfur and selenium present in high-purity arsenic cannot be removed easily from InAs and GaAs by zone refining. Three proposed methods for reducing the concentrations of these impurities in arsenic have been re-examined using tracer techniques. These are (*a*) vacuum sublimation, (*b*) sublimation in hydrogen, and (c) condensation into and distillation from a lead bath. Under favorable conditions, arsenic has been prepared containing the following atom fractions of sulfur, selenium, and tellurium, respectively: 1.1. x 10^{-9}, ~2 x 10^{-8}, and 2 x 10^{-8}.

Abstract No. 104

Preparation of High-Purity Gallium

Pierre de la Bretèque, Aluminium-Industrie-Aktien-Gesellschaft, Zurich, Switzerland

The preparation of high-purity gallium has been realized by purifying the sodium gallate in combination with a treatment of metallic gallium. Purification of sodium gallate is obtained by adding Na_2S; precipitation of the impurities in form of sulfides is favored by an addition of nickel salts. The metallic gallium is subjected to the following treatment: washing in an acid solution, filtering of the metal on porous plates, finally oxidation at elevated temperature by means of oxygen, followed by renewed filtering.

Abstract No. 105

Impurity Distribution of Various Doping Techniques in Floating Zone Crystal Growing

A. L. MacDonald and **E. Y. Wang,** Semiconductor Div., Raytheon Co., Newton, Mass.

In doping single-crystal silicon grown by floating zone techniques, one must use different doping procedures than commonly associated with the conventional Czochralski method. Several techniques have been investigated such as doping in the initial molten zone, using predoped rod, and reaction between molten zone and impurity vapor. Theoretical and experimental results for these methods are presented. The advantages for particular resistivity and impurity requirements are also discussed.

Abstract No. 106

Vibratory Polishing and Lapping of Semiconductor Materials

J. E. Cline and **S. J. Solomon,** Semiconductor Div., Raytheon Co., Newton, Mass.

The Syntron* vibratory polishing machine has been adapted for use in lapping of semiconductor materials and in polishing of device structures for metallographic examination. When conventional polishing techniques are used, alloyed semiconductor device structures present difficulties because of their small size, differing degrees of hardness, and brittleness of the semiconductor chip. It was found that the vibratory polisher in routine operation produces metallographic sections without scratches, rounding, or broken edges.

* Syntron Co., Homer City, Pa.

Abstract No. 107

Chemical Etching of Silicon, III. A Temperature Study in the Acid System

Harry Robbins and **Bertram Schwartz,** Hughes Products, Semiconductor Lab., Newport Beach, Calif.

Data showing the relationship between the etch rate of silicon and the temperature of the etchant are presented for several solutions composed of HF, HNO_3, H_2O, and $HC_2H_3O_2$. The activation energy of the reaction in solutions of various compositions is discussed in relation to the mechanism of the reaction.

Abstract No. 108

A Low Resistance Ohmic Contact for Silicon Semiconductor Devices

S. L. Matlow and **E. L. Ralph,** Hoffman Electronics Corp., Evanston, Ill.

A combination chemiplated and electroplated contact has been developed for use on silicon semiconductor devices. The chemiplating is done in an HF solution of $KAu(CN)_2$. The electroplating follow up can be with either a copper or gold solution. Data are given on the relationship between plating conditions and the resistance and bonding strength of the contact. A mechanism is proposed to explain the observed relationships.

Abstract No. 109

A Chemical Treatment for Rendering Silicon-Doped Aluminum Solderable

S. L. Matlow and **H. J. Gould,** Hoffman Electronics Corp., Evanston, Ill.

The use of aluminum alloying for making ohmic and rectifying contacts to silicon is well known in the art of the fabrication of silicon semiconductor devices. After alloying the aluminum is primarily in the form of the

α-phase (silicon doped). The various aluminum fluxes and solders available commercially do not work well on the α-phase. A set of chemical dip treatments is reported which render the α-phase solderable and give a strong bond to the aluminum.

Abstract No. 110

Depletion Layer Calculations for the Design of Diffused Semiconductor Devices

H. Lawrence and **R. M. Warner, Jr.**, Bell Telephone Labs., Inc., Murray Hill, N.J.

Diffused junctions in semiconductors generally exhibit linearly graded behavior at low voltage and step behavior at high voltage as far as depletion layer properties are concerned. These properties have been calculated for wide ranges of voltage and diffusion parameters for the case of a complementary error function distribution. The results delineate the ranges for which the two aproximations are valid and supply data for the extensive transition range where neither approximation is satisfactory. Results were obtained by solving Poisson's equation for the stated distribution analytically and by solving the equation specifying field continuity at the junction with the aid of the IBM 704.

Abstract No. 111

Development of Damage-Nucleated Structures in Silicon by Highly Preferential Etching

P. R. Pennington and **J. D. Turner**, Hughes Products, Semiconductor Lab., Newport Beach, Calif.

Study of the rates of etch by certain caustic etches on oriented silicon surfaces has revealed the existence of exceptionally high etch preferentiality. The rate of etch in a [111] direction is only a small fraction of the rates in directions normal to [111]. Structures with precisely controlled orientation and geometry may be etched in silicon with these preferential etches, their positions being determined by deliberately including damaged regions on the silicon surface.

Abstract No. 112

Silicon Transistors and Device Fabrication Using Damage-Nucleated Etched Structures

W. P. Waters and **P. Walker**, Hughes Products, Semiconductor Lab., Newport Beach, Calif.

Preferential caustic etching can be used to produce the crystallographically oriented structures required for fabrication of semiconductor devices. Precisely controlled damage of the surface nucleates a structure with reproducible penetration. Size and geometry is

determined by etch composition, etch time, and material orientation. Hexagonal, triangular, or rectangular pit forms and mesa structures are readily obtained. Hexagonal collector and emitter pits are used to produce webs of controlled one-half mil thickness. Silicon transistors are fabricated by aluminum evaporation.

Abstract No. 113

There is no
Abstract No. 113

Abstract No. 114

A New Approach to Semiconductor Surface Reactivity

G. A. Wolff, U. S. Army Signal Research and Development Lab., Fort Monmouth, N.J.

A surface study by combined physical, chemical, and crystallographic means leads to the following conclusions. The structure of clean surfaces of Si, Ge, SiC, and other semiconductors differs from the bulk structure because of abnormal bonding of the surface atoms. The normal bonding state is largely restored when halogens, chalcogens, or certain IIIb, Vb metals react with the surface atoms, "anion" and "cation" faces in III-V and II-VI compounds, and their bonding state are identified.

Abstract No. 115

Theory of Surface Conductance in Semiconductors

D. R. Frankl, Research Labs., Sylvania Electric Products Inc., Bayside, N. Y.

The Schrieffer theory of mobility of carriers in the space-charge layer near the surface of a semiconductor has been extended by eliminating an approximation in the form of the potential. The results are thus valid for accumulation as well as inversion layers and for carriers repelled from as well as attracted to the surface. Numerical results are presented for germanium and silicon.

Abstract No. 116

P-Layers* on Vacuum Heated Silicon

J. T. Law, T. M. Buck, and **F. G. Allen,** Bell Telephone Labs., Inc., Murray Hill, N.J.

Chemical p-layers have been found whenever etched silicon samples are heated in a Pyrex vacuum system to temperatures above 1300°K. By controlled etching these layers were found to extend 2-6 μ into the bulk. Experiments are described which show that the acceptor impurity is boron, originating from the Pyrex glass envelope. By using a quartz system, these layers have been eliminated. The electrically active residual impurities at the surface from all other sources are less than 10^6 cm^{-2}.

* See F. G. Allen, *J. Phys. Chem. Solids*, **8**, 120 (1959).

Abstract No. 117

Surface Channel Studies on Silicon P-N Junctions

Raymond Solomon and **P. O. Johnson,** Semiconductor Lab., Hughes Aircraft Co., Newport Beach, Calif.

Surface channels on silicon junctions have been investigated with a chopped light probe. The phase shift of the induced a-c photocurrent, relative to a standard signal, is measured as a function of distance of the light spot from the junction. The total phase shift along the surface may be as great as 360° and varies with surface preparation and ambient condition. It is observed that channels up to 20 mil in extent can exist even though the surface is not inverted. The properties of these anomalous channels are discussed in detail.

Abstract No. 118

Diffusion in Silicon Carbide

Hung-Chi Chang and **L. F. Wallace,** Westinghouse Electric Corp., Materials Engineering Depts., Pittsburgh, Pa.

The diffusion of acceptor and donor elements into silicon carbide has been performed in an open-tube carrier-gas system at temperatures from 1600° to above 2000°C. Techniques have been developed for measuring the diffusion constants based on the p-n junction depth and resistivity changes. Diffusion constants of aluminum in α-SiC have been investigated more quantitatively and are given by $D_{Al} = 1.8 \exp(-113{,}000/RT)$ for the [0001] direction with an average estimated error of $\pm$ 50%.

Abstract No. 119

Surface Concentration of Indium in Germanium from Gaseous Diffusion

K. M. Busen, E. L. Meeks, and **G. A. Shirn,** Sprague Electric Co., North Adams, Mass.

Impurities are indiffused into germanium from the vapor phase, using a specially designed closed system.

Correlations are established between the vapor pressure of the impurity and the concentration of the impurity on the germanium surface for various temperatures and times of the indiffusion cycle.

Abstract No. 120

Capillary Alloying: An Improved Alloying Method

K. M. Busen, J. J. Casey, Kurt Lehovec, and **A. C. Webb,** Sprague Electric Co., North Adams, Mass.

The alloying is done by placing a semiconductor wafer on the end of a capillary, and allowing the wafer to be in contact with a liquid metal or alloy contained in the capillary. The application of this procedure for diode and transistor technology is discussed and relations are given for calculating the alloying depth.

Abstract No. 121

Impurity Redistribution and Junction Formation in Silicon by Thermal Oxidation

M. M. Atalla and **E. Tannenbaum,** Bell Telephone Labs., Inc., Murray Hill, N. J.

In the process of growing an oxide on doped silicon, electrically active impurities near the Si/SiO_2 interface are redistributed depending on the diffusion coefficients and the distribution coefficient of the impurity between the oxide and the semiconductor. An analysis of this phenomenon predicts that single junction or two junction material can be obtained by oxidation of the surface of a compensated silicon crystal. For parabolic growth of the oxide, the surface concentration is independent of time, and the junction depth, gradient, and sheet resistivity vary with $t^{1/2}$. This has been demonstrated experimentally by oxidation of a compensated p-type silicon crystal doped with gallium and antimony.

Abstract No. 122

Metallurgical Analysis of the Bulk Diffusion Phenomenon in Silicon Power Transistors

P. X. Flaherty, Raytheon Co., Semiconductor Div., Newton, Mass.

In the analysis of bulk diffused silicon devices, it is necessary to use metallographic techniques other than conventional to determine basewidth thickness and junction condition. A bevel and stain technique in conjunction with the interferometric measuring methods has been employed successfully. Discussion of results and slides showing various facets of the bulk diffusion phenomenon are given.

Abstract No. 122A

Diffusion of Phosphorus into Silicon through an Oxide Layer

D. Kahng and **M. O. Thurston,** Electron Device Lab., The Ohio State University, Columbus, Ohio

The diffusivity of phosphorus in an amorphous silicon dioxide layer on silicon under oxidizing conditions has

been measured as a function of temperature and phosphorus concentration. The results have been used to predict quantitatively the phosphorus distribution in silicon for the case in which the sample is as free of oxide as possible initially, and the oxide grows during the diffusion. Masking effects in preoxidized silicon are also discussed theoretically and experimentally.

ELECTRO-ORGANIC

Abstract No. 123

Application of Constant Potential Electrolysis to the Process for Electrolytic Formation of Hydrazobenzene in Alkaline Emulsion. Mechanism for the Processes at Soluble and Insoluble Cathodes

Taro Sekine, Tadashi Seki, and **Kiichiro Sugino,** Lab. of Organic Electrochemistry, Dept. of Chemical Engineering, Tokyo Institute of Technology, Ookayama, Meguro-ku, Tokyo, Japan

To verify the proposed mechanism for hydrazobenzene formation at soluble cathode, constant potential electrolysis was carried out for each step of the reduction at Ni and Zn cathodes. At Ni, azoxybenzene and azobenzene could be reduced at about —1.25 v, (vs. S.C.E.) to hydrazobenzene, although the rate of the latter was very slow. At Zn, these reductions took place very smoothly at —1.50 ~ —1.57 v, at which the deposition of Zn^{++} ion occurs. The results in alcoholic alkaline solution are given for reference.

Abstract No. 124

Preparation of Crossed-Pinacols by Cathodic Reduction

M. J. Allen and **W. Pierson,** Research Dept., CIBA Pharmaceutical Products Inc., Summit, N.J.

The preparation of various crossed-pinacols by cathodic reduction of equimolar mixtures of two aromatic ketones is described. Also to be discussed is the apparent effect of the reference potential at the working electrode on the yield of crossed-pinacol.

Abstract No. 125

Kolbe Decomposition Potentials by Condenser and Ballistic Galvanometer

G. W. Thiessen and **F. F. Rawlings,** Monmouth College, Monmouth, Illl.

Decomposition potentials were determined for water and pyridine with Kolbe and other electrolytic solutes. Accepted values for non-Kolbe and Kolbe systems in water were obtained with no perceptible difference between uninhibited acetate and inhibited benzoate.

Evidence of a preliminary solvent process was found for aqueous systems. Pyridine revealed no solvent process and a higher decomposition potential for benzoate than acetate. Benzoate can be Kolbe discharged in pyridine. Consistent mechanisms for aqueous and nonaqueous Kolbe systems are presented.

Abstract No. 126

Electrolytic Formation of Oximes

Tadao Hayashi, Dept. of Chemistry, Ohio State University, Columbus 10, Ohio, and **C. L. Wilson,** Cranberry Lake, Sloatsburg, N.Y.

Reduction of an aqueous mixture containing an aliphatic or cycloaliphatic ketone and an inorganic nitrite or nitrate gave good yields of the ketoxime. Optimum conditions which gave yields of up to 70% are: *p*H 5.0, temperature, preferably below 15°, nitrite concentration of about 10%, and a rotating zinc cathode. Ketones used include acetone, methyl ethyl ketone, methyl isobutyl ketone, cyclopentanone, and cyclohexanone. Acetophenone gave no oxime and the reasons are discussed.

Abstract No. 127

The Stereo Selective Electrochemical Reduction of Nonconjugated Steroidal Ketones and Alpha Ketols

Peter Kabasakalian, Alvin Basch, James McGlotten, and **Milton Yudis,** Schering Corporation, Bloomfield, N.J.

Electrochemical reduction of a number of nonconjugated nuclear ketones at a stirred mercury cathode afforded the equatorial epimeric alcohol with a high degree of stereospecificity and in good yield comparable to reduction with sodium in alcohol. With α-hydroxy or acetoxy ketols. the hydroxyl function was cleaved reductively and the ketone reduced to an alcohol. Stereospecific reduction did not occur for the C_{20} side chain ketones but 50 to 70% of the C_{20} β-epimer was formed. The stereochemistry of electrochemical reduction of steroidal ketones is compared with chemical methods.

Abstract No. 128

Electrochemical Research in India and Japan

M. J. Allen, Research Dept., CIBA Pharmaceutical Products, Inc., Summit, N.J.

The Central Electrochemical Research Institute, in Karaikudi, India will be described together with a resumé of the basic and developmental research being done at this Institute. A discussion of the electrochemical work at Tokyo Institute oï Technology and Kyoto University will also be presented. Also to bè included is a description of the excellent instrumentation available in Japan for polarographic research. A film covering the trip will be shown.

Abstract No. 129

Polarographic Reduction of Some Acetylenic Linkages

A. H. Gropp and **J. C. Ramsey, Jr.**, Dept. of Chemistry, University of Florida, Gainesville, Fla.

Two series of unsaturated quaternary ammonium salts were prepared, each compound containing an acetylenic linkage. In one series the unsaturated group was held constant and the saturated groups varied; in the other series the saturated groups were held constant and the unsaturated group lengthened. It was found that increasing the size of the saturated groups lowered the polarographic reduction potential, while lengthening the unsaturated group increased the reduction potential.

Abstract No. 130

Polarographic Reductions of Benzyl Halides

L. W. Marple, L. E. I. Hummelstedt, and **L. B. Rogers,** Dept. of Chemistry and Laboratory for Nuclear Science, Massachusetts Institute of Technology, Cambridge 39, Mass.

The polarogram for benzyl chloride in a supporting electrolyte of lithium chloride has a dip in the limiting current due to electrostatic repulsion from the interface of the monovalent anion produced by electron transfer. The dip is decreased by the presence of electronegative substituents on the ring and is eliminated by a moderate concentration of surface-active tetramethylammonium ions in the solution.

Benzyl bromide and iodide each undergo two one-electron reduction steps. At concentrations greater than 10^{-4} M, adsorption phenomena arising from the reduction product, dibenzyl, are encountered.

Abstract No. 131

Polarography in Formamide, I. Some Transition Metal Ions, Zinc and Cadmium Ions

G. H. Brown and **Hsiao-shu Hsiung,** Dept. of Chemistry, University of Cincinnati, Cincinnati, Ohio

Polarographic data of certain transition metal ions, cadmium and zinc in formamide, have been obtained. The half-wave potentials (against a S. C. E.) were determined as follows: Cr −1.56, Mn −1.57, Fe −1.40, Co −1.20, Ni −0.98, Cu −0.12, Zn −1.07, Cd −0.69. All of these half-wave potentials were taken using 0.2M sodium perchlorate as the supporting electrolyte. Diffusion current constants of the ions studied in formamide were relatively small compared with other solvents; the difference is due mostly to the high viscosity of formamide.

Abstract No. 132

Polarographic Studies in Dimethylformamide VI. Reduction of Polyhalogenated Hydrocarbons

Stanley Wawzonek and **R. C. Duty,** Dept. of Chemistry, State University of Iowa, Iowa City, Iowa

The stability of carbanion intermediates in dimethylformamide suggested a study of the polarographic re-

duction of polyhalogenated hydrocarbons to determine whether carbenes would be formed as intermediates. Carbon tetrachloride was the only compound found to give a polarographic behavior which suggested the formation of a carbene. Three waves were obtained representing a reduction to chloroform, methylene chloride, and methyl chloride, respectively. The first wave was higher than the second and suggested the following steps.

$$CCl_4 + 2e \rightarrow :CCl_3 + Cl^-$$
$$:CCl_3 \rightarrow :CCl_2 + Cl^-$$
$$:CCl_2 + 2e \rightarrow :\ddot{C}Cl_2$$
$$:\ddot{C}Cl_2 + \text{solvent} \rightarrow CH_2Cl_2$$

Abstract No. 133

Millicoulometric Determination of Polarographic n-Values

Thos. De Vries, Dept. of Chemistry, Purdue University, Lafayette, Ind., and **J. L. Kroon,** Dow Chemical Co., Midland, Mich.

The microcoulometer, previously described, was used to determine polarographic n-values. The microcells held 0.3 - 0.7 ml of solution and the electrolysis time varied from 60 to 240 min. A dropping mercury electrode, with silver-silver chloride or silver iodide as reference electrode, was used. For aqueous solutions of 0.001M saccharin and hydrochloric acid as supporting electrolyte, an average n-value of 3.92 was obtained. The carbonyl and not the sulfanomide group of the molecule is reduced. For tetraiodophthalic anhydride in a buffered solution also containing 20% dimethylformamide, an average n-value of 8.0 was obtained, corresponding to the fission of the carbon-iodine bonds. For 2,2-dinitropropane in anhydrous ethyl alcohol, n-values of 1.4 to 2.2, instead of 4, were obtained. Apparently an intermediate compound, formed during the reduction step, is reoxidized at the anode to give the low results.

Abstract No. 134

Polarographic Reduction of Some Diketones

R. L. Flurry and **R. A. Day, Jr.,** Chemistry Dept., Emory University, Atlanta 22, Ga.

The polarographic reduction of p-diacetylbenzene, dibenzoylmethane, m-dibenzoylbenzene, 1,8-dibenzoylnaphthalene, 1,4-dibenzoylbutane has been studied in buffered ethanol-water solutions over a pH range of 2 to 13. Coulometric determinations of the number of electrons consumed under various conditions were run. Several products have been prepared by controlled potential electrolysis, and an attempt to explain the mechanism of reduction of these compounds is presented.

Abstract No. 135

Polarographic Reduction of Nonconjugated Steroidal Ketones

Peter Kabasakalian and **James McGlotten**, Schering Corp., Bloomfield, N.J.

The direct polarographic reduction of nonconjugated ketones in various positions both on the nucleus and the side chain of a steroid molecule has been carried out successfully. The polarographic reduction of hydroxyl and acetoxyl groups alpha to a carbonyl group is reported for the first time. The reduction wave of these groups in 20-ketosteroids merged with the reduction wave of the carbonyl group. In contrast, two distinct polarographic reduction waves were obtained with 16-hydroxy (or acetoxy)-17-ketosteroids.

Abstract No. 136

Electrolytic Reduction of Some Alkylnitrosamines

G. C. Whitnack, R. D. Weaver, and **H. W. Kruse,** Chemistry Div., Research Dept., U. S. Naval Ordnance Test Station, China Lake, Calif.

Polarographic reduction studies of some alkynitrosamines have been made in acidic, neutral, and alkaline solutions. Diffusion controlled waves were obtained and the mechanism of reduction is discussed. Data indicate that the course of reduction depends on *p*H and is rather complex in strong mineral acid. Good yields (50-75%) of 1,1-disubstituted hydrazines were obtained by large-scale electrolyses at a mercury cathode. Ammonia, alkylamine, nitrous oxide, and nitrogen were established as additional products of the reduction.

Abstract No. 136A

Rotating Cathode-Type Cell for the Preparation of Benzidine

K. S. Udupa, G. S. Subramanian, and **H. V. K. Udupa,** Central Electrochemical Research Institute, Karaikudi 3, S. India

The paper describes the work on the reduction of nitrobenzene in alkaline medium using rotating cathode. Conditions of electrolysis are similar to those established by Day and co-workers except that the reduction has been carried out employing higher current densities of the order of 15-20 amp/dm^2 as compared to 2 amp/dm^2. The reduction proceeds quite smoothly and almost quantitative yield of hydrazobenzene is obtained. A rotating disk-type m.s. cathode has been used along with cylindrical diaphragm of asbestos cloth supported on m.s. cage as anode chamber. The best conditions are (*a*) 10-20% alkali (NaOH) as catholyte and anolyte, (*b*) a depolarizer ratio of (w/v) 1:4, (*c*) current density of the order of 15-20 amp/dm^2, (*d*) temperature between 80°-85°C, (*e*) addition of 1% (w/v) of PbO to the catholyte. The paper also deals

with the method of isolation and recovery of the product and subsequent conversion to benzidine.

ELECTROTHERMICS AND METALLURGY

Abstract No. 137

Refractory Ceramic Coatings Today and Tomorrow

F. D. Shaw, Bettinger Corp., Milford, Mass.

This paper presents a review of present-day refractory coatings for protecting metals against oxidation erosion and abrasion at elevated temperatures. The more promising "super refractories" (carbides, nitrides, borides, and intermetallic cermets) are considered with respect to current developments in coating technology.

Abstract No. 138

The Adhesion of Vitreous Oxide Coatings to Metals

B. W. King and **W. H. Duckworth,** Battelle Memorial Institute, 505 King Ave., Columbus, Ohio

Results of an investigation of the fundamentals of the adherence of glasses to metals are presented. A distinction is made between the conditions essential for adherence and the phenomena related to adherence. Surface roughness was found not to be essential but to improve the apparent adherence when the bond was relatively weak. A theory is presented for the adherence of glasses to metals.

Abstract No. 139

A Liquid Phase Coating for Refractory Metals

G. D. Oxx, Jr., and **L. F. Coffin, Jr.,** Research Lab., General Electric Co., Schenectady, N.Y.

The concept of using a phase that is liquid at service temperatures as a component of coatings for refractory metals has been described. The liquid, an alloy of gold and silicon, is retained on a molybdenum surface by a capillary system made of molybdenum disilicide. The coating has the advantage of good thermal shock and has a self-healing characteristic.

Abstract No. 140

Testing Procedures for Evaluation of Coated Materials

J. L. Harp and **J. M. Allen,** Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio

Recent developments in aeronautics have focused attention on the behavior of materials under conditions of high heat flux and high temperature. Materials of interest must be protected from erosion and oxidation when exposed to a flow of high-temperature air; these materials and their coatings both must be thermal-shock resistant. Evaluation of bare and protected materials as heat sinks in rocket-motor tests indicates that

electrodeposited and flame sprayed coatings appear to be the most successful.

Abstract No. 141

Problems of Applying Metallic Coatings to Nonmetallic Bases

S. W. Bradstreet, Armour Research Foundation, Technology Center, Chicago 16, Ill.

(No abstract received)

Abstract No. 142

Coating Uranium Dioxide Powders with Metallic Films

J. H. Oxley, J. F. Hannah, and **J. M. Blocher, Jr.,** Battelle Memorial Institute, 505 King Ave., Columbus Ohio, and **I. E. Campbell,** National Steel Corp., Weirton, W. Va.

Conditions for tungsten coating of uranium dioxide powders by hydrogen reduction of tungsten hexachloride in a fluidized bed of the powder product have been established. The coated material should contain approximately 20 wt % tungsten to obtain essentially complete coverage of micron-sized uranium dioxide. The use of mechanical vibration was found to be necessary to obtain a satisfactory coating. Metals other than tungsten can be deposited on uranium dioxide powder particles and a number of such coated powders are now being explored for nuclear fuel applications.

Abstract No. 143

Silicon Carbide Coatings on Graphite Paper

J. G. Kemp, National Carbon Co., Fostoria, Ohio

(No abstract received)

Abstract No. 144

Metal-Ceramic Coatings for Protection of Graphite

W. L. Aves, Chance Vought Aircraft Co., Dallas, Texas

(No abstract received)

Abstract No. 145

Inorganic Coatings for Leading Edges of Boost-Glide Vehicles

H. A. Pearl, Bell Aircraft Co., Buffalo, N.Y.

(No abstract received)

Abstract No. 146

Coatings for Structural Aircraft Materials

D. H. Leeds, Douglas Aircraft Co., Santa Monica, Calif.

(No abstract received)

Abstract No. 147

Refractory Coatings for Ramjet and Supersonic Aircraft Applications

S. Sklarew, Marquardt Aircraft Co., Van Nuys, Calif.
(No abstract received)

Abstract No. 148

Protective Coatings for Medium Temperature Ranges

A. R. Stetson, Solar Aircraft Corp., San Diego, Calif.
(No abstract received)

Abstract No. 149

Reactions in the Niobium-Hydrogen System

W. M. Albrecht, W. D. Goode, and **M. W. Mallett,** Battelle Memorial Institute, 505 King Ave., Columbus, Ohio

Equilibria and elevated-temperature x-ray studies show that a solid solution of hydrogen in niobium is produced throughout most of the system. A miscibility gap was found at low temperatures and pressures with a critical point at about 140°C, 0.01 mm of mercury hydrogen pressure, and 0.3 H/Nb. Sorption rates at 300° to 550°C were initially linear. At higher temperatures, sorption rates were controlled by diffusion in the metal matrix. Diffusion coefficients at 600° to 700°C can be expressed by:

$$D = 0.0215 \exp [\ (-9370 \pm 600)/RT]$$

Desorption rates were slower than those predicted by diffusion.

Abstract No. 150

A Study of the Iodide Niobium Process

R. F. Rolsten, Eastern Lab., E. I. du Pont de Nemours and Co., Inc., Gibbstown. N.J.

It was established that the rate of deposition of niobium by the thermal decomposition of niobium iodides can be expressed in units of moles of niobium deposited per unit length of a decomposition element per unit of time. The rate of deposition of niobium was studied for deposition temperatures from 750° to 1100°C, feed temperatures from 240° to 470°C, and with grades of crude niobium having different surface areas.

Abstract No. 151

The Behavior of Rhenium in Electron Tube Environments

G. B. Gaines, C. T. Sims, and **R. I. Jaffee,** Battelle Memorial Institute, Columbus, Ohio

Rhenium, tungsten, and rhenium-coated tungsten have been exposed to carburizing atmospheres and to

water-cycle attack, rhenium and tungsten to aluminum oxide at 1750°C for 7000 hr, and rhenium to boron-containing atmospheres. It was confirmed that rhenium, unlike most other refractory metals, does not form a carbide. Rhenium forms a boride, which forms a eutectic with another boride, or with rhenium, and melts under 2250°C. Thoriated rhenium maintains microstructural and mechanical integrity after long exposure in vacuum at 1750°C in contact with alumina. Rhenium and rhenium-coated tungsten are highly resistant to water-cycle effects.

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INDEX TO AUTHORS

(The number in the right hand column is the abstract number used in the Technical Program and in the Abstracts.)

	Abstract No.
Albrecht, W. M.	149
Alderuccio, C. L.	80
Allen, F. G.	116
Allen, J. M.	140
Allen, M. J.	124, 128
Amlie, R.	11
Angstadt, R. T.	22
Arnold, E.	95
Atalla, M. M.	121
Aves, W. L.	144
Bartlett, J. H.	70, 71
Basch, Alvin	127
Bate, R. T.	85
Bauman, H. F.	7
Bergishagen, Finn	82
Black, J.	95
Blocher, J. M., Jr.	142
Bockstie, L.	17
Boudart, M.	59
Boyd, H. J.	78
Bradstreet, S. W.	141
Brattain, W. H.	42
Brenner, Abner	65, 75
Breslin, J. T.	91
Bride, J. E.	81
Brodd, R. J.	5
Brown, G. H.	131
Bruins, P. F.	26
Buck, T. M.	47, 116
Burbank, Jeanne	12
Busen, K. M.	119, 120
Button, M. J.	96
Cabrera, N.	46
Cahan, B. D.	11, 22
Cahoon, N. C.	1, 4, 9
Campbell, I. E.	142
Caplan, D.	35
Casey, J. J.	120
Champness, C. H.	92
Chang, Hung-Chi	118
Chessin, H.	80
Cline, J. E.	106
Coffin, L. F., Jr.	139
Cohen, M.	35, 36
Couch, D. E.	65
Davenport, W. H.	38
Day, R. A., Jr.	134
DeCrescente, M. A.	37

	Abstract No.
de la Bretèque, Pierre	104
DeLap, J. H.	6
De Vries, J.	15
De Vries, Thos.	133
Dewald, J. F.	50
DeWane, H. J.	2, 5
Dirkse, T. P.	13, 14, 15
Ditzenberger, J. A.	103
Dodson, V. H.	21
Draley, J. E.	31
Duckworth, W. H.	138
Duty, R. C.	132
Eischens, R. P.	60
Eisenberg, M.	7
Engell, H. J.	51
Falk, S. Uno	27
Farnsworth, H. E.	45
Faust, C. L.	81
Faust, J. W., Jr.	54
Fink, M. S.	89
Fischler, A. S.	101
Flaherty, P. X.	122
Flannery, R. J.	66
Flurry, R. L.	134
Francis, H. T.	79
Frankl, D. R.	115
Gaines, G. B.	151
Gale, R. H.	37
Garrison, W. E., Jr.	64
Gatos, H. C.	58, 96
Gaule, G. K.	91
Gerischer, H.	49
Giles, F. H., Jr.	70, 71
Gillet, I. E.	67
Gilman, J. J.	48
Glicksman, R.	19, 20
Goode, W. D.	149
Gould, H. J.	109
Graham, A. H.	77
Graham, A. K.	78
Green, Milton	88
Greenberg, I. N.	88
Greenberg, Sherman	31
Gropp, A. H.	129
Gurklis, J. A.	81
Gwathmey, A. T.	62
Hackerman, Norman	55
Handler, Paul	44
Hannah, J. F.	142
Harman, T. C.	97
Harp, J. L.	140
Hartley, A. M.	64
Harvey, A.	35
Hauffe, K.	61

(Index cont'd)

Abstract No.

Hayashi, Tadao 126
Holmes, M. R. 82
Hsiung, Hsiao-shu 131
Hummelstedt, L. E. I. 130

Jaffee, R. I. 151
Jenkins, L. H. 39
Johnson, P. O. 117
Johnson, R. S. 1
Jones, E. L. 23, 24
Juretschke, H. J. 43

Kabasakalian, Peter 127, 135
Kahng, D. 122A
Kalhammer, F. 16
Kaminsky, George 93
Katz, M. J. 89
Kemp, J. G. 143
King, B. W. 138
King, C. V. 57
Kohan, L. R. 79
Kolb, K. E. 63
Kordesch, K. 10
Kornfeil, Friedrich 3
Korver, M. P. 1
Krivanek, F. J. 4
Kroon, J. L. 133
Kruse, H. W. 136

Lacombe, Paul 52
Lander, J. J. 25
Lasser, M. 16, 17
Lavine, M. C. 96
Law, J. T. 116
Lawless, K. R. 62
Lawrence, H. 110
Lee, C. A. 93
Leeds, D. H. 146
Lehovec, Kurt 120
Lichter, B. D. 29
Lippincott, T. W. 63
Lorking, K. F. 74
Loverude, T. H. 18
Lozier, G. S. 8
Lumley, E. J. 84
Lyons, V. J. 100

MacDonald, A. L. 105
Maier, H. B. 68
Makrides, A. C. 33, 34
Mallett, M. W. 149
Mark, H. B. Jr. 6
Marko, A. 10
Marple, L. W. 130
Marvel, C. S. 64
Mason, D. R. 102

(Index cont'd)

	Abstract No.
Matlow, S. L.	108, 109
McGlotten, James	127, 135
McGraw, L. D.	81
Meeks, E. L.	119
Morehouse, C. K.	8, 19
Nelson, E. E.	25
Newton, E. H.	73
Nole, V. F.	38
Ockerman, J. B.	11
Olshefski, P. J.	86
Oxley, J. H.	142
Oxx, G. D., Jr.	139
Pastore, J. R.	91
Paquin, R. A.	76
Pearl, H. A.	145
Pennington, P. R.	111
Petrocelli, J. V.	56
Pierson, W.	124
Pinkerton, H. L.	78
Pochop, C. G.	91
Powell, A. S.	37
Rairden, J. R.	30
Ralph, E. L.	108
Ramsey, J. C., Jr.	129
Rawlings, F. F.	125
Read, H. J.	77
Reese, W. E.	101
Reynolds, D. C.	98
Robbins, Harry	107
Robertson, W. D.	38
Rogers, L. B.	130
Rolsten, R. F.	150
Rothberg, M.	87
Rosenberg, A. J.	94
Rüetschi, Paul	11, 22
Ruther, W. E.	31
Ryan, N.	84
Salkind, A. J.	26
Santoro, P. F.	37
Saubestre, E. B.	69
Schaefer, H. F.	9
Schwartz, Bertram	107
Seiger, H. N.	28
Seki, Tadashi	123
Sekine, Taro	123
Sewell, P. B.	36
Shaw, F. D.	137
Sherwood, G. R.	82
Shirn, G. A.	119
Shombert, D. J.	85, 86
Shuttleworth, R. A.	91

(Index cont'd)

	Abstract No.
Silvestri, V. J.	100
Silvey, G. A.	100
Simon, A. C.	23, 24
Sims, C. T.	151
Sklarew, S.	147
Solomon, Raymond	117
Solomon, S. J.	106
Soule, D. E.	90
Stern, Milton	32, 33, 40, 41
Stetson, A. R.	148
Stockbridge, C. D.	36
Strauss, A. J.	99
Struthers, J. D.	103
Subramanian, G. S.	136A
Sugino, Kiichiro	123
Sullivan, F.	73
Swann, Sherlock, Jr.	64
Tannenbaum, E.	121
Thiessen, G. W.	125
Thomas, J. E. Jr.	66
Thomassen, Lars	102
Thurston, M. O.	122A
Tragert, W. E.	30
Trivich, D.	66
Turner, D. R.	53
Turner, J. D.	111
Turner, W. J.	101
Udupa, H. V. K.	136A
Udupa, K. S.	136A
Vosburgh, W. C.	6
Waber, J. T.	34A
Wagner, Carl	29
Wales, C. P.	12
Walker, P.	112
Wallace, L. F.	118
Wang, E. Y.	105
Ward, J. W.	34A
Warner, R. M., Jr.	110
Warschauer, D. M.	98
Waters, W. P.	112
Wawzonek, Stanley	132
Weaver, R. D.	136
Webb, A. C.	120
Weil, Rolf	76
Weingarten, I. R.	85, 86, 87
Weissberger, S.	95
Whelan, J. M.	103
Whitnack, G. C.	136
Wiers, Brandon	13
Wilson, C. L.	63, 126
Wissenberg, Herman	40, 41
Wolff, G. A.	114
Wood, G. B.	83
Woodberry, P. T.	72
Yudis, Milton	127
Zaromb, S.	16, 17

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